SITE SPECIFIC QUALITY ASSURANCE PROJECT PLAN FOR LONG-TERM MONITORING AND MAINTENANCE PROGRAM PFAS SAMPLING ADDENDUM

U.S. ARMY GARRISON FORT DEVENS
DEVENS, MASSACHUSETTS

OCTOBER 2017

Prepared for:
U.S. Army Corp of Engineers
New England District
Concord, Massachusetts

Prepared by:
Renova Environmental Services.
Contract No.: W912WJ-15-C-0038

NOTICE

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QAPP Worksheet #1 - Title and Approval Page
(UFP-QAPP Section 2.1)

Site Name/Project Name: Long-Term Monitoring and Maintenance Program
Site Location: U.S. Army Garrison Fort Devens, Devens, MA

Document Title: Long-Term Monitoring and Maintenance Program UFP Quality Assurance Project Plan – PFAS Sampling Addendum

Lead Organization: U.S. Army Corps of Engineers

Preparer's Name and Organizational Affiliation: Stacey Felts-Bock, Renova Environmental Services, LLC

Preparer's Address, Telephone Number, and E-mail Address: 3417 Sunset Ave, Ocean Township, NJ 07712, 732-659-1000, pheyman@renovaenviro.com

Preparation Date (Day/Month/Year): 18 October 2017

Investigative Organization's Project Manager/Date: 10/19/2017
Signature
Printed Name/Organization: Stacey Felts-Bock, Renova Environmental Services

Steven Passafaro Digitally signed by Steven Passafaro DN: cn=Steven Passafaro, o=Sovereign Consulting Inc., ou, email=spassafaro@sovcon.com, c=US Date: 2017.10.18 19:04:29 -04'00'
Investigative Organization's Project QA Officer/Date:
Signature
Printed Name/Organization: Steven Passafaro, Sovereign Consulting Inc.

REDDY.PENELOPE.W.1502105066 Digitally signed by REDDY.PENELOPE.W.1502105066 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USA, cn=REDDY.PENELOPE.W.1502105066 Date: 2017.10.18 22:33:25 -04'00'
Lead Organization's Project Manager/Date:
Signature
Printed Name/Organization: Penelope Reddy, U.S. Army Corps of Engineers

QAPP Worksheet #2 - QAPP Identifying Information
(UFP-QAPP Section 2.2.4)

Site Name/Project Name: Long-Term Monitoring and Maintenance Program
Site Location: U.S. Army Garrison Fort Devens, Devens, MA
Site Number/Code: N/A
Operable Unit: N/A
Contractor Name: Renova Environmental Services
Contractor Number: W912WJ-15-C-0038

Title: PFAS Sampling Addendum
Revision Number: 0
Revision Date: October 2017

Contract Title: Environmental Services
Work Assignment Number: Delivery Order 003

1. Identify regulatory program: Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), Superfund Amendments and Reauthorization Act of 1986 (SARA), Resource Conservation and Recovery Act (RCRA), and National Contingency Plan (NCP) programs
2. Identify approval entity: USEPA Region 1
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. List dates of scoping sessions that were held: 6 October 2015
5. List dates and titles of QAPP documents written for previous site work, if applicable:
   June 2016 Site Specific QAPP for Annual Long-Term Monitoring and Maintenance Program (Renova, 2016)
6. List organizational partners (stakeholders) and connection with lead organization:

   U.S. Army Corps of Engineers (USACE), U.S. Army Environmental Command (AEC), USEPA Region 1, Massachusetts Department for Environmental Protection (MassDEP)
7. List data users: USACE, U.S. Army, USEPA, MassDEP, Renova Environmental Services, Sovereign Consulting Inc.
8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

   Worksheet #16 - Project Schedule/Timeline Table - The project schedule is provided to USACE under separate cover.

   Worksheet #18 - Sampling Locations and Methods/SOP Requirements Table - Sample locations will be presented in the project specific field sampling plan (FSP) which will reference this QAPP for analytical methods and SOP requirements.

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Project Management and Objectives		
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2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	Worksheet #5 Worksheet #6 Worksheet #7 Worksheet #8 Site Safety and Health Plan and Accident Prevention Plan Lab ELAP Accreditation / MassDEP Certification
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	Worksheet #9 Worksheet #10 FSP
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	Worksheet #11 Worksheet #12

QAPP Worksheet #2
QAPP Identifying Information
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Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
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2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	Worksheet #14 Site-Specific WP, FSP
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QAPP Identifying Information
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Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
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QAPP Worksheet #2
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Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
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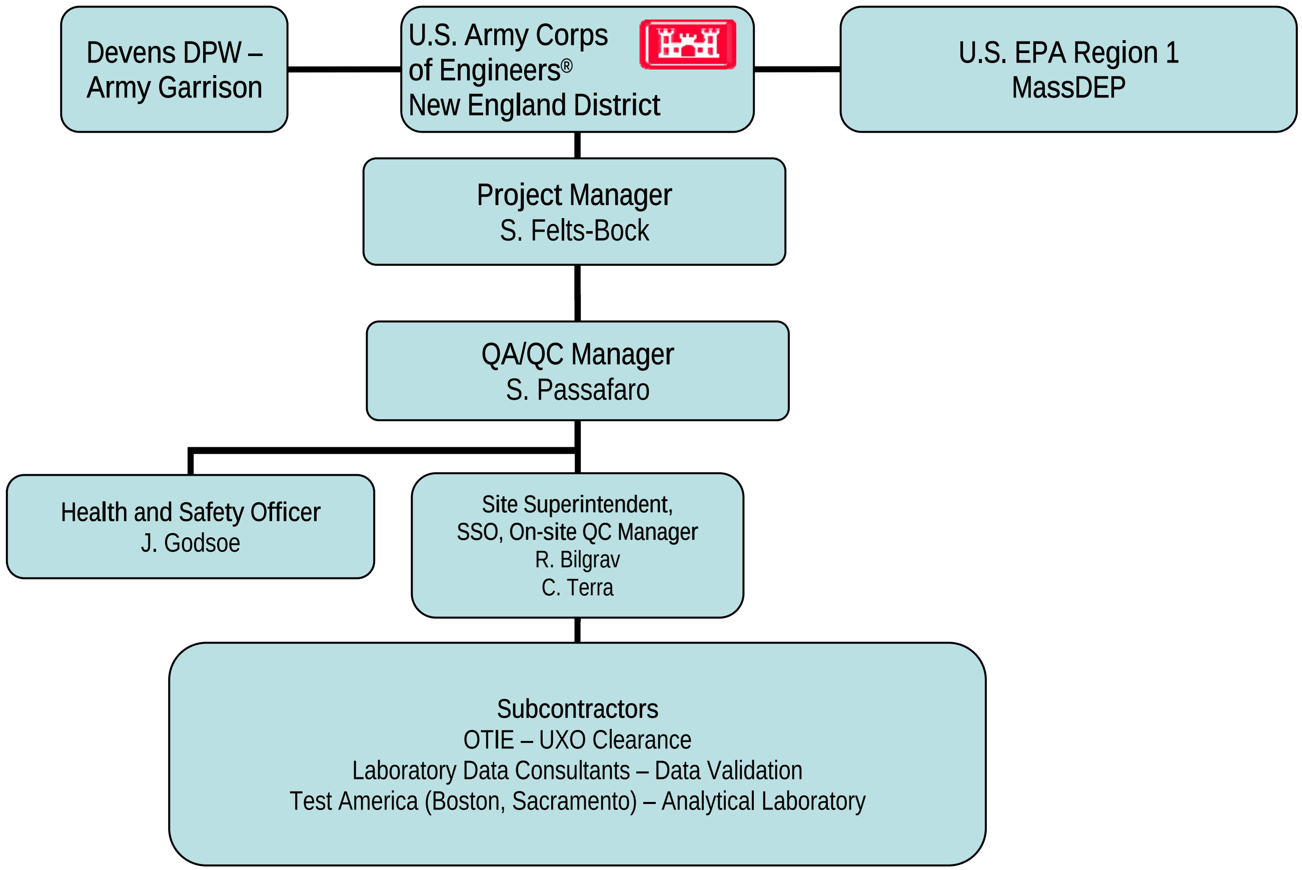
QAPP Worksheet #3 - Distribution List
(UFP-QAPP Manual Section 2.3.1)

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	Document Control Number
Penelope Reddy	Technical Lead	USACE	978-318-8160	Penelope.Reddy@usace.army.mil	QAPP-01
Yixian Zhang	Project Chemist	USACE	978-318-8730	yixian.zhang @usace.army.mil	QAPP-01
Ray Prisk, PE	Director of Public Works	U.S. Army – DPW	978-796-3665	Raymond.a.prisk.civ@mail.mil	QAPP-01
Benjamin Rice	Environmental Division Chief	U.S. Army – DPW	978-796-2096	benjamin.j.rice2.civ@mail.mil	QAPP-01
Paulo Heyman	President, Program Manager	Renova Environmental Services	732-659-1000	pheyman@renovaenviro.com	QAPP-01
Stacey Felts-Bock, PE	Project Manager	Renova Environmental Services	732-659-1000	Stacey@renovaenviro.com	QAPP-01
Steven Passafaro, PE	QA/QC Manager	Sovereign Consulting	508-339-3200	spassafaro@sovcon.com	QAPP-01
Shauna McKellar	Project Chemist	Laboratory Data Consultants	760-827-1100	scuenco@lab-data.com	QAPP-01
Jerry Lanier	Project Manager	Test America – Savannah	912-354-7858	Jerry.lanier@testamericainc.com	QAPP-01
Robert Hrabak	Operations Manager	Test America – Sacramento	916-373-5600	Robert.Hrabak@testamericainc.com	QAPP-01

QAPP Worksheet #4 - Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Stacey Felts-Bock	Project Manager	732-659-1000		
Steven Passafaro	QA/QC Manager	508-339-3200		
Shauna McKellar	Project Chemist – LDC	760-827-1100		
Jerry Lanier	Project Manager – Test America	912-354-7858		

QAPP Worksheet #5 - Project Organizational Chart
(UFP-QAPP Manual Section 2.4.1)



QAPP Worksheet #6 - Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
USACE Point of Contact	USACE	Penelope Reddy	978-318-8160	USACE Project Manager. Point of contact between USACE and Contractor.
Management of all Project Phases	Contractor Project Manager	Stacey Felts-Bock	732-659-1000	Renova liaison with USACE. Communicates with all Contractor staff for all project issues.
Field Corrective Issues; changes in scope of field work or QAPP amendments	QA/QC Manager	Steven Passafaro	508-339-3200	The need for Field Corrective Action, changes in scope of field work, or QAPP amendments will be determined by the QA/QC Officer who will report directly to the Contractor Project Manager. The Contractor PM will then communicate changes to USACE verbally and by e-mail within 48 hours. If warranted, the Contractor PM will send concurrence letter to USACE for review and concurrence prior to implementing scope change. USACE will notify all stakeholders of changes as needed.
Release of Analytical Data	Project Chemist	Shauna McKellar	760-827-1100	The Project Chemist oversees all data validation activities and releases data for project use after validation and verification upon authorization by the Contractor PM.
Analytical data quality issues	Test America PM Project Chemist	Jerry Lanier Shauna McKellar	912-354-7858 760-827-1100	The Laboratory PM (or designate) or Project Chemist will notify (verbally or via e-mail) the Contractor Project Manager within two business days when an issue related to laboratory data is discovered. The Laboratory PM is responsible for coordinating with their subcontracted laboratories any corrective actions and project data quality objectives.

QAPP Worksheet #7 - Personnel Responsibilities and Qualification Table
(UFP-QAPP Manual Section 2.4.3)

Name	Title	Organizational Affiliation	Responsibilities
Penelope Reddy	Technical Lead	USACE	Oversees project implementation, including scoping, document review, and data evaluation.
Yixian Zhang	Project Chemist	USACE	Participates in data review, evaluation, and review of the QAPP.
Stacey Felts-Bock	Project Manager	Renova	Serves as single point of contact to USAEC/Army representatives. Manages and ensures adherence to schedule, performance milestones, and budgets. Tracks and monitors cost and schedule and issues weekly and monthly cost/schedule reports as necessary. Approves final reports.
Steven Passafaro, PE	QA/QC Manager	Sovereign	Assists the Contractor PM in all duties. Ensures quality aspects of the project are implemented, documented, and maintained. Reviews work plans, technical reports, fact sheets, and other documents.
Julie Godsoe	Health and Safety Officer	Sovereign	Develops and implements the Health and Safety program for the project. Develops APP/ Site Specific SSHP. Conducts safety training. Performs safety audits and implements corrective actions, as necessary. Suspends work for unsafe conditions. Approves changes to APP/SSHPs. Maintains accident and safety incident investigation reports.
Shauna McKellar	Project Chemist	LDC	Coordinates laboratory-related QA/QC functions with laboratory. Oversees data validation and quality assurance review.
Jerry Lanier	Project Manager	Test America	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with project staff. Coordinates project analytical requirements with their subcontracted laboratories.

QAPP Worksheet #8 - Special Personnel Training Requirements Table
(UFP-QAPP Manual Section 2.4.4)

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Activities	OSHA 40 Hour HAZWOPER with annual 8 hour refreshers, First Aid Training	Certified Training Professionals	NA	Field Operations Personnel	Renova, Sovereign	Corporate Offices
Site Supervision	OSHA 8 Hour Site Supervisor	Certified Training Professionals	NA	Site Supervisor	Renova, Sovereign	Corporate Offices
Analytical Chemistry	DoD Environmental Laboratory Accreditation Program (ELAP) Accreditation	Primary Accrediting Body	NA	NA	Test America	Laboratory Offices
Analytical Chemistry	Massachusetts Certification	Massachusetts	NA	NA	Test America	Laboratory Offices

1 – Current training certificates will be maintained in a file at the primary office location for each employee performing work at each jobsite where training is required for the position assignment.
2 – Additional training/certification requirements are listed in the project health and safety plan.

QAPP Worksheet #9 - Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Environmental Services **Projected Date(s) of Sampling:** October 2015 - September 2020 **Project Manager:** Paulo Heyman	**Site Name:** U.S. Army Garrison Fort Devens **Site Location:** Devens, Massachusetts

Date of Session: 6 October 2015
Scoping Session Purpose: Contract Kick-Off Meeting; Project Planning

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Penelope Reddy	Technical Lead	USACE	978-318-8160	Penelope.Reddy@usace.army.mil	USACE Project Manager
Ray Prisk, PE	Director of Public Works	U.S. Army – DPW	978-796-3665	Raymond.a.prisk.civ@mail.mil	DPW Point of Contact
Anne MacMillan	Environmental Scientist	Bluestone Environmental Group	610-647-9500	amacmillan@bluestoneenviro.com	DPW Contractor
Paulo Heyman	Program Manager	Renova Environmental Services	732-659-1000	pheyman@renovaenviro.com	Program Manager
Marc Cicalese	QA/QC Manager	Sovereign Consulting	973-219-3049	mcicalese@sovcon.com	QA/QC Manager
Steven Passafaro, PE	Project Manager	Sovereign Consulting	508-339-3200	spassafaro@sovcon.com	Project Manger

Comments/Decisions: The scoping session was conducted prior to implementation of the project and preparation of the QAPP. The agenda for the scoping session is provided below.

I. **Introduction & Purpose**
   a. Roles and Responsibilities/Introductions
   b. Project Overview

II. **Project Schedule**

III. **Fall 2015 Field Sampling Event**
   a. AOC 26
   b. South Post Monitoring Wells
   c. Utilization of Existing Documents (QAPP, LTMMP, HASP) to perform Fall event in November/December

IV. **Submittals**
   a. Upfront Documents (PMP, QAPP, HASP, LTMMP)
   b. 2015 Annual Report
   c. Schedule of Values

V. **Action Items**

QAPP Worksheet #10 - Problem Definition
(UFP-QAPP Manual Section 2.5.2)

Problem Definition

The South Post-U.S. Army Garrison Fort Devens is a range facility used to provide training to active, reserve, and National Guard units and local, state, and federal police. Over time activities at the range have resulted in contamination of the groundwater with metals, explosives and perchlorate. Currently, the Army is required to complete long-term monitoring (LTM) and annual reporting within the South Post Impact Area (SPIA) of South Post at the following sites and/or areas: (1) annually at Area of Concern (AOC) 26; (2) biannually at AOC 27; and (3) annually at select South Post Monitoring (SPM) Wells.

In addition, the USEPA has identified per- and polyfluoroakyl substances (PFAS) as emerging contaminants and in January 2009 established provisional health advisory levels (HALs) for perfluorooctane sulfonate (PFOS), which is used in aqueous film forming foams to extinguish fires, and perfluorooctanoic acid (PFOA), which is used in a variety of consumer products. In May 2016, the USEPA then issued a lifetime drinking water HAL for both PFOS and PFOA following toxicity studies (USEPA 2016a and 2016b). In order to confirm/deny the presence of PFAS in SPIA groundwater and, if present, whether it is migrating from the South Post, the USEPA requested as part of their comments to the 2016 Annual Report (USEPA, 2017b) that groundwater samples are collected for PFAS analysis from seven existing perimeter groundwater monitoring wells (SPM-93-06X, SPM-93-08X, SPM-93-10X, SPM-93-12X, SPM-93-16X, SPM-97-23X, and SPM-97-24X) located throughout SPIA.

Project Description

The environmental services to be performed as part of this project will include the collection of groundwater samples from seven existing SPIA monitoring wells for PFAS analysis. Detailed information regarding the scope of services described above will be outlined within the project-specific FSP. It is important to note that because SPIA is an active range and there is the potential for UXO, range control will be coordinated with in advance of the work, and a UXO technician will be present and accompanying field workers at all times to clear the path to and from monitoring wells.

QAPP Worksheet #11 - Project Quality Objectives /Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

Who will use the data? The primary data users will be USACE and the U.S. Army Environmental Command (AEC); the secondary users will include USEPA Region I, MassDEP, Renova, and Sovereign.
What will the data be used for? The data will be used by Renova and Sovereign to prepare a PFAS Investigation Report. USACE and the U.S. Army will use the data and the conclusions of the post sampling investigation report to to confirm/deny the presence of PFAS in SPIA groundwater and, if present, whether it is migrating from the South Post. If PFOA and PFOS compounds are not present above the HAL, no further evaluation will be required. If compounds are above HAL, further evaluation may be required to determine potential receptors, the source of PFAS compounds, and background concentrations.
What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques) Specific data and analytical data requirements will be presented in the project specific FSP which will reference this QAPP for analytical methods and SOP requirements.
How "good" do the data need to be in order to support the environmental decision? The data must be of comparable quality to the LTM Program data collected in past sampling events and of sufficient quality to support evaluation of results against the requirements of the ROD and HALs to allow for an accurate evaluation of the LTM optimization options.
How much data are needed? (number of samples for each analytical group, matrix, and concentration) The number of samples for each analytical group, matrix, and concentration will be presented in the project specific FSP. Specific analyses are listed on QAPP Worksheet #19.
Where, when, and how should the data be collected/generated? This information will be presented in the project specific FSP.
Who will collect and generate the data? Renova and its subcontractor, Sovereign, will collect the samples and will generate field data (e.g., water-level measurements and water quality parameters). Test America will generate the analytical data for PFAS analysis.
How will the data be reported? Laboratory data will be reported in analytical packages (produced in .PDF format) that will, at a minimum, contain all necessary information to allow for validation in accordance with the EPA Tier II protocols. The laboratory will J qualify results below the LOQ, where applicable. The laboratory will produce SEDD Stage 2a deliverables or higher, consistent with DOD QSM valid values that have been screened against the ADR.Net project eQAPP provided by LDC. Reviewed SEDD files and reviewed ADR text files will be provided for import into USACE Environmental Database Management System (EDMS).
How will the data be archived? Complete project file records will be maintained in Renova's office and will be updated under the PM's direction. Project records will be maintained during the lifespan of the contract, and all data and project files will be provided to the USACE throughout the project and again upon project closeout.

QAPP Worksheet #12 - Measurement Performance Criteria Tables
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table

Matrix	Aqueous
Analytical Group	PFAS
Laboratory	Test America - Sacramento
Concentration Level	All

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
See FSP	537 (Modified) / L-1	Precision	RPD <30%	Field Duplicates	S & A
		Accuracy/Bias	< LOQ	Blanks (Field, Equipment)	S & A
		Accuracy/Bias	No target compounds ≥1/2 LOQ	Method Blanks	A
		Accuracy/Bias	%R DoD 5.0 Limits	LCS, MS	A
		Precision	%RPD analyte specific, DoD 5.0 Limits	LCSD, MSD	A
		Accuracy/Bias	%R DoD 5.0 Limits	Surrogate Compounds	A
		Accuracy/Bias	Initial and continuing calibration standards within standards specified by the laboratory SOP	Initial and continuing calibration standards	A
		Sensitivity	Initial calibration acceptance limits specified by the laboratory SOP	LOQ set at low level calibration standard	A
		Sensitivity	Limit of detection (LOD) must produce a response at least 3 times greater than instrument noise level	LOD studies	A

QAPP Worksheet #13 - Secondary Data Criteria and Limitations
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
LTM Program	Previous Annual Reports for South Post Impact Area Long Term Ground Water Monitoring (Various originating organizations and dates)	SPIA groundwater sampling summary and analytical data	Compared to groundwater analytical results to identify spatial and temporal trends at the SPIA	None, except as identified for individual data points in the associated DQE

QAPP Worksheet #14 - Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

Sampling Tasks: All field operations and sampling will be conducted in accordance with the project specific FSP.
Analysis Tasks: Chemical analysis will be performed by a subcontracted laboratory, Test America. Test America is DoD Environmental Laboratory Program (ELAP) accredited and holds MassDEP certifications for analytes where available. Analyses will be performed in accordance with the analytical methods specified in Worksheet #19. The contract laboratory will strive to meet the PALs, quantitation limits, and detection limits (DLs), as shown in Worksheet #15. Test America will perform chemical analyses that will follow laboratory-specific SOPs (Worksheet #19).
Quality Control Tasks: The type and frequency of field QC samples are summarized in Worksheet #20.
Secondary Data: See Worksheet #13.
Data Management Tasks: After each sampling/monitoring event is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratories will meet the requirements of the technical specification. Electronic data deliverables (EDD) will be in the SEDD Stage 2a or higher format.
Documentation and Records: Data packages are tracked at the laboratory by assignment of sample delivery group (SDG) numbers. The laboratory sends PDF formats of all data packages. Data packages are recorded, tracked, and stored at secure on-site and off-site electronic storage locations.
Assessment/Audit Tasks: Renova personnel perform assessments and internal audits of sampling and analysis processes. These audits consist of systems (e.g., field sampling and laboratory inspections) and performance (e.g., analysis of QA split samples) audits. External audits of sampling procedures and laboratory processes by USACE or MassDEP may also be conducted.
Data Review Tasks: The Laboratory Data Consultant, Inc. (LDC) Automated Data Review software (ADR.Net) will be used to review the analytical data. The LDC project chemist will review the ADR.Net report for all data generated for the project. The laboratory will produce SEDD Stage 2a or higher deliverables, consistent with DOD QSM valid values that have been screened against the ADR.Net project eQAPP provided by LDC. Additional details on data validation are presented in Worksheets #34 through #36.
Corrective Action: Corrective actions will be implemented when deficiencies are identified. Corrective actions will be initiated and implemented in conjunction with the PM, QC Manager, and Project Chemist.

QAPP Worksheet #15 - Reference Limits and Evaluation Tables
(UFP-QAPP Manual Section 2.8.1

Reference Limits and Evaluation Table

Matrix: Aqueous
Analytical Group: PFAS by 537 (Modified)
Laboratory: Test America
Concentration Level: All

Analyte	CAS Number	Project Action Limit	Project Quantitation Limit	Achievable Laboratory Limits		
				DLs	LODs	LOQs
Perfluorohexanoic acid (PFHxA)	307-24-4	NA	2.50	0.786	2.00	2.50
Perfluoroheptanoic acid (PFHpA)	375-85-9	NA	2.50	0.802	2.00	2.50
Perfluorooctanoic acid (PFOA)	335-67-1	70	2.50	0.748	2.00	2.50
Perfluorononanoic acid (PFNA)	375-95-1	NA	2.50	0.654	2.00	2.50
Perfluorodecanoic acid (PFDA)	335-76-2	NA	2.50	0.440	1.00	2.50
Perfluoroundecanoic acid (PFUnA)	2058-94-8	NA	2.50	0.748	2.00	2.50
Perfluorododecanoic acid (PFDoA)	307-55-1	NA	2.50	0.584	2.00	2.50
Perfluorotridecanoic Acid (PFTriA)	72629-94-8	NA	2.50	0.551	2.00	2.50
Perfluorotetradecanoic acid (PFTeA)	376-06-7	NA	2.50	0.400	1.00	2.50
Perfluorobutanesulfonic acid (PFBS)	375-73-5	NA	2.50	0.918	2.00	2.50
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	NA	2.50	0.870	2.00	2.50
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	70	4.00	1.28	3.00	4.00
N-ethyl perfluorooctane sulfonamidoacetic acid (NEtFOSAA)	2991-50-6	NA	20.0	5.02	15.0	20.0
N-methyl perfluorooctane sulfonamidoacetic acid (NMeFOSAA)	2355-31-9	NA	20.0	5.64	15.0	20.0
13C2 PFHxA	STL00993	NA	100	50.0	100	100
13C4 PFOA	STL00990	NA	100	50.0	100	100
13C5 PFNA	STL00995	NA	100	50.0	100	100
13C2 PFDA	STL00996	NA	100	50.0	100	100
13C2 PFUnA	STL00997	NA	100	50.0	100	100
13C2 PFDoA	STL00998	NA	100	50.0	100	100
18O2 PFHxS	STL00994	NA	100	50.0	100	100
13C4 PFOS	STL00991	NA	100	50.0	100	100
13C4-PFHpA	STL01892	NA	100	50.0	100	100

Notes:
1) All units are ng/L.
2) Achievable DLs, LODs, and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
3) Project Action Limits shown are USEPA Health Advisory Levels as specified in the May 2016 USEPA Health Advisories for PFOS and PFOA (EPA 822-R-16-004 & EPA 822-R-16-005).

QAPP Worksheet #16 - Project Schedule Timeline
(UFP-QAPP Manual Section 2.8.2)

The project schedule is provided to USACE within the project specific FSP.

QAPP Worksheet #17 - Sampling Design and Rationale
(UFP-QAPP Section 3.1.1)

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach): The general sampling approach rational for the PFAS sampling is to collect groundwater samples from select monitoring wells at South Post as requested by the EPA in order to confirm/deny the presence of PFAS in SPIA groundwater and, if present, whether it is migrating from the South Post (USEPA, 2017b). Specific sampling details and procedures are provided in the project-specific FSP.
Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations): The number of samples to be collected, the sampling frequency, the sampling locations, and the matrices to be sampled will be specified within the project specific FSP. Analytical groups are specified on Worksheet #15.

QAPP Worksheet #18 - Sampling Locations and Methods/SOP Requirements

(UFP-QAPP Manual Section 3.1.1)

Sample locations for PFAS sampling will be presented in the project specific FSP which will reference this QAPP for analytical methods and SOP requirements.

QAPP Worksheet #19 - Analytical SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Aqueous	PFAS	All	537 (Modified)/L-1	250 ml	2 x 250 ml HDPE (Note – Teflon lined caps not to be used.)	Cool 2°C-6°C (Note – samples should be shipped in their own cooler to avoid possible cross-contamination.)	14 days until extraction/ 40 days extraction to analysis

QAPP Worksheet #20 - Field Quality Control Sample Summary Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations [1]	No. of Field Duplicate Pairs	No. of MS/MSD	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab [1]
Aqueous	PFAS	All	L-1	TBD	1 per 20 samples	1 MS & 1 MSD per 20 samples	1 per 20 samples	1 per 20 samples	0	TBD

[1]Sampling locations and number of samples are included in the FSP.

QAPP Worksheet #21 - Project Sampling SOP References Table
(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
SOP-GW-001	Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells, Revision 3, 19 January 2010	EPA	See SOP	N	None
SOP #2043	Manual Water Level Measurements 11 February 2000	EPA	See SOP	N	None
SOP #2006	Sampling Equipment Decontamination	EPA	See SOP	N	None
SOP-PFC-01	Field Sampling Protocols to Avoid Cross-Contamination at Perfluorinated Compounds (PFCs) Sites	DoD	See SOP	N	None

For all other field sampling operating procedures, see FSP.

QAPP Worksheet #22 - Field Equipment Calibration, Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
YSI, Horiba	Calibration Verification	Per Manufacturer's Instructions	Per Manufacturer's Instructions	Visual	Daily - Before and after each use	Manufacturer's Instructions	Operator Correction or Replacement	Field Technician	Manufacturer's Instructions
Photoionization Detector (PID)	Calibration Verification	Per Manufacturer's Instructions	Per Manufacturer's Instructions	Visual	Daily - Before and after each use	Manufacturer's Instructions	Operator Correction or Replacement	Field Technician	Manufacturer's Instructions
Water Level Meter	Calibration Verification	Per Manufacturer's Instructions	Per Manufacturer's Instructions	Visual	Daily Visual Inspection; Annual Calibration by Vendor	+/- 0.01 foot	Operator Correction or Replacement	Field Technician	Manufacturer's Instructions

Note: 1) Rental equipment and instruments will be used in the field. The rental firms will be responsible for the proper care, maintenance, and repair of these items, and for tracking and documenting equipment and instrument maintenance and repairs. The Site Superintendent will ensure that the equipment is operational and suitable for its intended use and will conduct and record daily pre-use and post-use calibration checks.

QAPP Worksheet #23 - Analytical SOP References Table
(UFP-QAPP Manual Section 3.2.1)

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
L-1	Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue) [Method 537 (Modified)], Revision 2.7, 9/20/17	Definitive	PFAS	LC/MS/MS	Test America – Sacramento	N

QAPP Worksheet #24 - Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
LC/MS/MS	ICAL	Prior to sample analysis or when CCV fails	r ≥ 0.995 or %RSD ≤20%	Perform maintenance, Recalibrate, Prepare new standards; Reanalyze impacted samples	Analyst	L-1
	ICV	Following initial calibration; midpoint of the calibration	%D≤30%			
	CCV	After every 10 samples and at the beginning and end of an analytical sequence	%D≤30%			

ICAL – Initial Calibration; ICV – Initial Calibration Verification; CCV – Continuing Calibration Verification

QAPP Worksheet #25 - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
LC/MS/MS	Check pressure and gas supply daily – change when <200 pounds per square inch (psi), change analytical column as needed, change mobile phase when insufficient for run or contamination, change inlet filters as needed for contamination. Clean the source and replace the filaments.	PFAS	Check pump pressure, check for leaks, check for adequate mobile phase.	Source cleaning is performed when the instrument response deteriorates. Other instrument maintenance is done as needed to keep the instrument performing at peak performance.	CCV < 30%	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	L-1

QAPP Worksheet #26 - Sample Handling System
(UFP-QAPP Manual Appendix A)

For a full discussion of sample handling requirements and methods, see the project specific FSP. A basic reference is provided below.

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Technicians
Sample Packaging (Personnel/Organization): Field Technicians
Coordination of Shipment (Personnel/Organization): Field Technicians
Type of Shipment/Carrier: Contract Carrier or Hand Delivery
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Receipt Personnel / Test America
Sample Custody and Storage (Personnel/Organization): Sample Management Personnel / Test America
Sample Preparation (Personnel/Organization): Extractions or Digestion Department Personnel / Test America
Sample Determinative Analysis (Personnel/Organization): Analyst / Test America
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from data reporting, approximately 75 days from sample collection.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days
Biological Sample Storage (No. of days from sample collection): Not Applicable
SAMPLE DISPOSAL
Personnel/Organization: Test America
Number of Days from Analysis: 60 days

QAPP Worksheet #27 - Sample Custody Requirements
(UFP-QAPP Manual Section 3.3.3)

Field Sample Handling and Chain-of Custody (COC) Procedures

Custody of samples must be maintained and documented at all times to ensure the integrity of a sample from collection through analysis. An accurate written record is necessary to trace the possession and handling of the sample. This documentation is referred to as the "COC" form. COC begins when samples are collected in the field, and is maintained by storing the samples in secure areas until custody can be passed on. All samples will be delivered to the laboratory accompanied by a COC form that will describe the sample identifiers, the analytical parameters, and the persons who are responsible for the sample integrity.

After sample collection has been completed and the caps have been placed back on each bottle, ample containers will be labeled with the sample location number, sampler's name, date, sample preservation, and analytical fraction.

Following collection, samples will be placed on ice in a secure cooler and attended by field personnel or placed in locked vehicles or designated storage areas until analysis or shipment to the off-site laboratory. The samples will then be shipped to the laboratory in coolers packed with ice and bubble wrap or equivalent packing material, to cushion the samples to prevent breakage and to maintain the required temperature for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure the chain-of-custody is maintained. Samples will be shipped to the laboratory by an overnight courier to ensure that maximum sample holding times are not exceeded or they will be picked up directly by the laboratory via laboratory courier. The maximum allowable sample holding times are presented in Worksheet #19. This worksheet also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain sample integrity.

Each sample collected will be assigned a unique sample tracking number, as described in the FSP. The sample number, sample collection date and time, person collecting the sample and a list of the sample analyses to be performed will be recorded on each container, and also on the chain-of-custody form. The chain-of-custody form is a two-part form: the original copy accompanies the samples to the analytical laboratory, and the second copy will be archived in the project files.

The following information will be recorded on the chain-of-custody form:

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- QC sample designation (as applicable)
- Analysis method
- Special handling instructions
- Destination of samples
- Name, date, time and signature of each individual releasing custody

Laboratory Custody Procedures

The laboratory sample custodian will inspect the integrity of the cooler custody seals and measure the temperature of the samples received using the "Temperature Blank" container included in each cooler. The samples will be checked against the chain-of-custody form for holding times, sample identification, and integrity. The samples will be logged into the laboratory management system. Custody of the samples will be maintained and recorded in the laboratory, from receipt to analysis, and this record will be included with the data package deliverables.

QAPP Worksheet #28 - QC Samples Tables
(UFP-QAPP Manual Section 3.4)

Field QC Samples Table

Matrix	Aqueous					
Analytical Group	All					
Concentration Level	All					
Sampling SOP	FSP					
Analytical Method/ SOP Reference	All					
Sampler's Name	TBD					
Field Sampling Organization	Renova					
Analytical Organization	Test America					
No. of Sample Locations	TBD					
QC Sample:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Cooler Temperature Blanks	1 per Cooler	4^0C, ± 2^0C, or as stated in Worksheet #19	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy/bias-Preservation	4^0C, ± 2^0C, or as stated in Worksheet #19
Field Duplicates	1 per 20 field samples of similar matrix	See Worksheet 12	Qualify data as needed	Renova Personnel	Precision	See Worksheet 12
MS/MSD	1 per 20 field samples	See Worksheet 12	Qualify data as needed	Lab / Renova Personnel	Accuracy/Bias	%R Lab generated limits
Field/Equipment Blank	1 per 20 field samples	<LOQ	Qualify data as needed	Renova Personnel	Accuracy/Bias	<LOQ

Lab QC Samples Table

Matrix	Aqueous
Analytical Group	All
Concentration Level	All
Sampling SOP	FSP
Analytical Method/ SOP Reference	All
Sampler's Name	TBD
Field Sampling Organization	Renova
Analytical Organization	Test America
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/extraction batch	< ½ LOQ	Locate source of contamination, correct problem, re-extract and analyze associated samples	Analyst	Accuracy/bias (contamination)	< ½ LOQ
Calibration Blank	Before beginning a sample run, after every 10 samples, and at the end of the sequence	< ½ LOQ	Locate source of contamination, correct problem, re-analyze calibration blank and previous ten samples	Analyst	Accuracy/bias (contamination)	< ½ LOQ

Lab QC Samples Table

Matrix	Aqueous
Analytical Group	All
Concentration Level	All
Sampling SOP	FSP
Analytical Method/ SOP Reference	All
Sampler's Name	TBD
Field Sampling Organization	Renova
Analytical Organization	Test America
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	1/extraction batch	See Worksheet 12	Evaluate exceedance and impact on sample data. Re-extract batch if necessary	Analyst	Accuracy	See Worksheet 12
Limit of Detection (LOD)	Quarterly	LODs must produce a response greater than 3 times the instrument noise level; using standard concentration within 2-3 times the detection limit.	Check for errors. Repeat LOD study, if necessary	Analyst	Sensitivity	LOD must produce a response greater than 3 times the instrument noise level

Lab QC Samples Table

Matrix	Aqueous
Analytical Group	All
Concentration Level	All
Sampling SOP	FSP
Analytical Method/ SOP Reference	All
Sampler's Name	TBD
Field Sampling Organization	Renova
Analytical Organization	Test America
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Prior to analyzing samples	See Worksheet 24	Perform maintenance; Re-calibrate; Prepare new standards	Analyst	Accuracy	See Worksheet 24
Continuing Calibration verification (CCV)	See Worksheet 24	See Worksheet 24	Correct problem; re-run CCV. Repeat ICAL, if necessary. Re-analyze all samples since last successful ICAL or CCV.	Analyst	Accuracy	See Worksheet 24
Independent Calibration Check (ICV) standard	Once after each initial calibration; prior to sample analysis	See Worksheet 24	Correct problem and verify ICV. If that fails, repeat ICAL.	Analyst	Accuracy	See Worksheet 24

Lab QC Samples Table

Matrix	Aqueous
Analytical Group	All
Concentration Level	All
Sampling SOP	FSP
Analytical Method/ SOP Reference	All
Sampler's Name	TBD
Field Sampling Organization	Renova
Analytical Organization	Test America
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate Spike	All field and QC samples.	%R must be within DoD QSM acceptance limits	Check calculations for errors, check instrument performance, re-extract and re-analyze the samples if the above show no problems or flag the data if sample matrix interference is present.	Analyst, Supervisor	Accuracy/Bias	%R must be within DoD QSM acceptance limits

Lab QC Samples Table

Matrix	Aqueous
Analytical Group	All
Concentration Level	All
Sampling SOP	FSP
Analytical Method/ SOP Reference	All
Sampler's Name	TBD
Field Sampling Organization	Renova
Analytical Organization	Test America
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standard	Every field and QC sample prior to analysis	The response must not deviate by more than 50% from the average response of the ICAL and the most recent CCV.	Inspect instrument for malfunctions and correct. Reanalyze corresponding samples.	Analyst, Supervisor	Accuracy/Bias	The response must not deviate by more than 50% from the average response of the ICAL and the most recent CCV.

QAPP Worksheet #29 - Project Documents and Records Table
(UFP-QAPP Manual Section 3.5.1)

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Logbook Field Sample Forms Chain-of-Custody Records Sampling Notes Photographs	Equipment Calibration Logs Field Data Records Tracking Records Field Instrument Maintenance Logs	Sample receipt/login form Sample storage records Sample preparation logs Equipment calibration logs Sample analysis run logs Reported results for standards, QC checks, and QC samples Data completeness checklists Telephone logs Extraction/clean-up records Laboratory EDD ERIS or ERIS convertible EDD Raw data	Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted)	Health and Safety Plan Quality Assurance Project Plan FSP Project Planning Documents Permits Site Maps

QAPP Worksheet #30 - Analytical Services Table
(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number
Aqueous	PFAS	All	TBD	See Worksheet 23	Data packages within 3 weeks	Test America - Boston 240 Bear Hill Road, Suite 104 Waltham, MA 02451 Jerry Lanier 912-354-7858 Test America – Sacramento 880 Riverside Parkway West Sacramento, CA 95605 916-373-5600	N/A

NOTE:
1) No backup laboratories are planned at this time. If any are selected, they will be sub-contracted to Test America, the Prime laboratories.
2) Data packages will be provided in PDF format and will be Level III/IV CLP-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data).
3.) Field sampling crews shall deliver samples to the closest prime laboratory (i.e. Test America Laboratory in Waltham, MA). Test America will then ship samples under chain of custody to the DoD ELAP accredited laboratory branch as applicable.

QAPP Worksheet #31 - Planned Project Assessments Table
(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Laboratory System Audit	Every 2 years	Ext.	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Test America	Test America	DoD ELAP Accrediting Body Auditor
Field Audit	Annually	Int.	Renova	QA/QC Officer	Renova Project Manager	Field Personnel	QA/QC Officer

QAPP Worksheet #32 - Assessment Findings and Corrective Action Responses
(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Laboratory System Audit	Written audit report	Jerry Lanier, PM Test America	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP
Field Assessments	Memo or email	Steven Passafaro, QA/QC Officer, Sovereign	Verbal notification next business day; written within 1 week	Corrective action report	Steven Passafaro, QA/QC Officer, Sovereign	As soon as possible depending on nature of deficiency
Data Review and Validation	Validation Report	Shauna McKellar, Project Chemist, LDC	Within 28 days of receipt of laboratory report	Written notifications of findings to the laboratory	Jerry Lanier, PM Test America	As soon as possible

QAPP Worksheet #33 - QA Management Reports Table

(UFP-QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Analytical Data Review Report	Per Sample Delivery Group	See FSP	LDC Data Validator	QA/QC Officer or key technical resource lead for study area
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon resolution of problem (same day)	Test America PM	Renova PM, Project file
Final Project Reports	As data is compiled and interpreted	See FSP	Renova PM	USACE-NAE

QAPP Worksheet #34 - Verification (Step I) Process Table
(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain-of-Custody Forms	The Site Superintendent or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file.	Internal	Site Superintendent – Renova
	The Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Renova QA/QC Officer will then check that the chain-of-custody form was signed/dated by the Site Superintendent or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Test America 2 – QA/QC Manager, Renova
QAPP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed project samples listed in the QAPP tables and field logs have been collected.	Internal	QA/QC Manager or designee, Renova
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	QA/QC Manager or designee, Renova
Sample coordinates	Verify that actual sample locations are correct and in accordance with the QAPP proposed locations. Document any discrepancies in the final report.	Internal	QA/QC Manager or designee, Renova
QAPP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	QA/QC Manager or designee, Renova

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
QAPP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables / DOD QSM Version 5.0	Ensure that all laboratory SOPs and DOD QSM Version 5.0 were followed. Verify that the correct analytical methods/SOPs were applied. Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory PM will contact the Renova PM via telephone or e-mail for guidance prior to report preparation.	External	Laboratory PM, Test America
QAPP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	QA/QC Manager or designee, Renova
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory PM will sign the case narrative for each data package.	External	Laboratory PM, Test America
EDDs/ Analytical Data Packages	Each EDD will be verified against the chain-of-custody and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the DL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from data by the automated data review module (ADR) in EDMS. Further, data will be reviewed by a professional to manually edit qualifiers if needed prior to importing to EDMS.	External	Data Validator PM, LDC
	Each data package will be verified for completeness by the Data Validator. Missing information will be requested by the validator from the Laboratory PM.	External	Data Validator PM, LDC

QAPP Worksheet #35 - Validation Steps IIa and IIb) Process Table
(UFP-QAPP Manual Section 5.2.2)

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and store at the required temperature and sample pH (or not acidified) for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Data Validator, LDC
IIa/IIb	QAPP/ Laboratory Data Packages/ EDDs	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Data Validator, LDC
		Automated review of quality control parameter, surrogates, holding time, field QC, field duplicates, MS/MSD, method blank, and LCS/ LCSD, if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12 and in method SOPs.	
		Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The data validator will verify that elements of the data package are present, and if not, the laboratory will be contacted and the missing information will be requested. ADR will be performed as per Worksheet #36.	
IIb	QAPP/Laboratory Data Packages /EDDs	Summarize deviations from methods, procedures, or contracts in the Automated Data Review Report. ADR will qualify the data results based on MPC and list QC sample deviation. The qualified data will be assigned a reason code that will explain any data qualifications.	Data Validator, LDC

QAPP Worksheet #36 - Analytical Data Validation (Step IIa and IIb) Summary Table
(UFP-QAPP Manual Section 5.2.2)

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Aqueous	All[1]	All	EPA National Functional Guidelines for Organic Superfund Data Review (January 2017) for parameters evaluated by ADR.net.[2]	LDC

Note
1) All data packages will undergo automated data review. The following parameters/QC checks will be evaluated by ADR.net: surrogates, holding time, field blanks, field duplicates, MS/MSD, method blank, and LCS/ LCSD, if available. The following data qualifiers will be used: Non-detect (U), Non-detect and estimated (UJ), rejected or unusable (R), or estimated (J).

2) A modified outline will be used for validation since CLP methods are not being used for this project.

QAPP Worksheet #37 - Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics: ***Completeness***: The QA/QC Officer or designee will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the PM determines that the deviations compromise the ability to meet project objectives they will consult with the COR and other project team members, as necessary (determined by the COR), to develop appropriate corrective actions. ***Precision***: The Project Chemist will determine whether off-site laboratory analytical precision goals for field duplicates and laboratory duplicates were met for field test kit or other field test method QC split samples. ***Accuracy***: The Project Chemist will determine whether the accuracy/bias goals were met for on-site and off-site analytical project data. This will be accomplished by comparing percent recoveries of MS/MSD, LCS, and/or LCSD to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination, instrument calibration variability, matrix spike, matrix spike duplicate, and laboratory control samples. ***Representativeness***: The QA/QC Officer or designee and Project Chemist will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and analyzed in accordance with this QAPP and by reviewing spatial and temporal data variations. Further off-site analytical data will be evaluated for potential cross contamination by evaluation of analytical blanks and the equipment blank. ***Comparability***: The Project Chemist will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. Select on-site field test kit and/or field test method samples will be split and analyzed by the off-site laboratory. A correlation between the on-site and off-site lead results will be completed. ***Sensitivity***: The Project Chemist will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared.
Describe the evaluative procedures used to assess overall measurement error associated with the project: After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. The Project Chemist will assess whether the data collectively support the attainment of project objectives. Project precision, accuracy, comparability, representativeness, completeness, and sensitivity will be evaluated.

Identify the personnel responsible for performing the usability assessment: The PM, Project Chemist, and QA/QC Officer will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Project Team. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The data will be reviewed per Worksheet #36. Significant findings from the ADR will be summarized in the project reports. If any systemic matrix bias or analytical bias is found, then that will be discussed in the project report along with the limitations on the data. Comparison of field test kit and/or field test method results to off-site QC split samples will be presented a combined frequency of correct decision making and linear correlation graph. Graphical presentations of the investigation data such as concentration tag maps will be generated as part of the overall data evaluation process.

REFERENCES

Department of Defense 2013. *DoD Quality Systems Manual for Environmental Laboratories*. Version 5.0. July.

Department of the Navy 2015. Perfluorinated Compounds Interim Guidance/Frequently Asked Questions. January.

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USEPA, 2002. *Guidance for Quality Assurance Project Plans* USEPA QA/R-5, USEPA QA/G-5, Quality Assurance Manuals.

USEPA, 2005. *Uniform Federal Policy for Quality Assurance Plans*.

USEPA 2014a. *Emerging Contaminants Fact Sheet – Perfluorooctane Sulfonate (PFOS) and Perflourooctanoic Acid (PFOA). Solid Waste and Emergency Response*. EPA 505-F-14-001. March.

USEPA 2016a. *Drinking Water Health Advisory for Perfluorooctane Sulfonate (PFOS)*. EPA 822-R-16-004. May.

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USEPA, 2017a. *USEPA National Functional Guidelines for Superfund Organic Methods Data Review (SOM02.4)*. January.

USEPA, 2017b. *Comments to Fort Devens Reserve Force Training Area 2016 Annual Report, Long-Term Monitoring, South Post Impact Area*. 6 June.

Appendix A

TestAmerica Savannah
5102 LaRoche Avenue

Savannah, GA 31404-6019
phone 912.354.7858 fax 912.352.0165

Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

TestAmerica Laboratories, Inc.

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	_______ of ______ COCs
Address	**Analysis Turnaround Time**			Sampler:
City/State/Zip	☐ CALENDAR DAYS ☐ WORKING DAYS			**For Lab Use Only:**
(xxx) xxx-xxxx Phone	TAT if different from Below _________			Walk-in Client:
(xxx) xxx-xxxx FAX	☐ 2 weeks			Lab Sampling:
Project Name:	☐ 1 week			
Site:	☐ 2 days			Job / SDG No.:
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)																	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____________

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ Return to Client ☐ Disposal by Lab ☐ Archive for _________ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (^{o}C): Obs'd:_________ Corr'd:__________ Therm ID No.:____________

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Form No. CA-C-WI-002, Rev. 4.6, dated 09/02/2015

Appendix B

Title: Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue

[Method 537 (Modified)]

Approvals (Signature/Date):

Robert Hrabak Technical Manager	9/19/17 Date	Joe Schairer Health & Safety Manager / Coordinator	9/19/17 Date
Lisa Stafford Quality Assurance Manager	9/20/2017 Date	Crystal Pollock Laboratory Manager	9.20.17 Date

Facility Distribution No. Uncontrolled **Distributed To:** Sacramento Bids Folder

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water, soil, sediment, and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
Perfluoro-n-hexanoic acid	PFHxA	307-24-4
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluoro-n-decanoic acid	PFDA	335-76-2
Perfluoro-n-undecanoic acid	PFUdA (PFUnA)	2058-94-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8
Perfluoro-n-tetradecanoic acid	PFTeDA (PFTA)	376-06-7
Perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid	PFODA	16517-11-6
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octanesulfonamide	FOSA	754-91-6
N-ethylperfluoro-1-octanesulfonamide	EtFOSA	4151-50-2
N-methylperfluoro-1-octanesulfonamide	MeFOSA	31506-32-8
Perfluorinated sulfonamidoacetic acids (FOSAA)		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9
Fluorotelomer sulfonates (FTS)		
1H,1H,2H,2H-perfluorohexane sulfonate *(4:2)*	4:2 FTS	757124-72-4
1H,1H,2H,2H-perfluorooctane sulfonate *(6:2)*	6:2 FTS	27619-97-2
1H,1H,2H,2H-perfluorodecane sulfonate *(8:2)*	8:2 FTS	39108-34-4

Abbreviations in parenthesis are the abbreviations listed in Method 537, where they differ from the abbreviation used by the laboratory's LIMS.

1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	250 mL	2.0 ng/L – 100 ug/L	2.0 ng/L - 400 ug/L
Soil/Sediment	5 g	0.2 ug/kg – 20 ug/kg	0.2 ug/kg - 100 ug/kg
Tissue	1g	1.0 ug/kg – 100 ug/kg	1.0 ug/kg – 500 ug/kg

1.3. The procedure for the analysis of water samples via in line solid phase extraction (SPE) for a subset of the list in Section 1.1 using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500 is described in Attachment 1 of this SOP.

1.4. This procedure also includes direction for preparing and analyzing samples to determine "Total Oxidizable Precursors", which may assist in improving understanding of potential PFAS environmental risk.

1.5. When undertaking projects for the Department of Defense (DOD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Water samples are extracted using a solid phase extraction (SPE) cartridge, unless EtFOSA and MeFOSA are requested. PFAS are eluted from the cartridge with an ammonium hydroxide/methanol solution.

2.2. Soil/sediment/tissue samples are extracted with a KOH/methanol solution using an orbital shaker for 3 hours followed by sonication for 12 hours. The mixture is centrifuged and the solvent filtered.

2.2.1. Optional cleanups may include sample freezing and/or cleanup by SPE cartridge, unless EtFOSA and MeFOSA are requested.

2.3. The final 80:20 methanol:water extracts are analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using 20 mM ammonium acetate/water and methanol. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFAS.

2.4. An isotope dilution technique is employed with this method for the compounds of interest. The isotope dilution analytes (IDA) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs, or deuterated analogs of the compounds of interest, and they are spiked into the samples at the time of extraction. This technique allows for the correction for analytical bias encountered when analyzing more chemically complex environmental samples. The isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have an identically labeled analog are quantitated by the IDA method using a closely related labeled analog.

2.5. Quantitation by the internal standard method is employed for the IDA analytes/recoveries. Peak response is measured as the area of the peak.

2.6. Samples for the "Total Oxidizable Precursor" assay (TOP) are analyzed in two phases – an aliquot is prepared and analyzed as a normal sample, and a second aliquot is subjected to oxidation with potassium persulfate and sodium hydroxide prior to solid phase extraction and analysis. The total perfluorocarboxylic acid value is determined for each aliquot, and the difference calculated.

3. DEFINITIONS

3.1. PFCAs: Perfluorocarboxylic acids

3.2. PFSAs: Perfluorinated sulfonic acids

3.3. FOSA: Perfluorinated sulfonamide

3.4. PFOA: Perfluorooctanoic acid

3.5. PFOS: Perfluorooctane sulfonic acid

3.6. MPFOA: Perfluoro-n-[1,2,3,4-13C4]octanoic acid. Carbon-13 labeled PFOA

3.7. MPFOS: Perfluoro-1-[1,2,3,4-13C4]octanesulfonic acid. Carbon-13 labeled PFOS

3.8. PTFE: Polytetrafluoroethylene (e.g., Teflon®)

3.9. SPE: Solid phase extraction

3.10. PP: Polypropylene

3.11. PE: Polyethylene

3.12. HDPE: High density polyethylene

3.13. IDA: Isotope dilution analyte

3.14. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

4.3. PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.

4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

4.3.2. Random evaporation losses have been observed with the polypropylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.

4.3.3. Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the same teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.

4.4. Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

4.5. Commercial sources of PFOS, PFHxS, PFOA, and other PFAS may produce several peaks in the chromatogram. These adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the peaks matches the retention time of the single labeled linear PFAS peak. In general, earlier peaks are branched isomers and are not a result of peak splitting. When reference standards of technical mixtures of specific PFAS are available, they should be used to ensure that all appropriate peaks are included during peak integration. Refer to Section 7, Reagents, for the available technical mixtures utilized by this SOP.

4.6. The phenomenon of the linear and branched isomers of PFOS exists for other PFAS, such as PFHxS, PFBS, PFOA, EtFOSAA, and MeFOSAA. Thus, in an attempt to reduce PFOS bias, it is required that *m/z* 449>80 transition be used as the quantitation transition.

4.7. Both branched and linear PFAS can potentially be found in the environment. For the compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard and/or sample must be integrated and the areas included.

4.8. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.15 ng/L or 0.01 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns

5.1.1. Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS must be handled in the laboratory as hazardous and toxic chemicals.

5.1.2. Exercise caution when using syringes with attached filter disc assemblies.

Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Eye protection that satisfies ANSI Z87.1 (as per the TestAmerica Corporate Safety Manual), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.

5.1.6. The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed, or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.7. Glass containers are not to be used for “tumbling” soil samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide (3-0-0)	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent damage, including blindness. Brief exposure to 5000 PPM can be fatal.
Hexane (2-3-0)	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid (3-0-1)	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.
Methanol (2-3-0)	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Potassium Hydroxide (3-0-1)	Corrosive Poison		Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Potassium Persulfate (2-0-1-OX)	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Sodium Hydroxide (3-0-1)	Corrosive Poison	2 mg/cm^3 (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. 15 mL polypropylene test tubes with polypropylene screw caps.

6.2. 50 mL graduated plastic centrifuge tubes.

6.3. 125 mL HDPE bottles with HDPE screw caps.

6.4. 250 mL HDPE bottles with HDPE screw caps.

6.5. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.

6.6. Extract concentrator or nitrogen manifold with water bath heating to 50-55°C.

6.7. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.

6.8. 300 µL autosampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent.

6.9. SPE columns

6.9.1. Phenomenex Strata SPE C18, 6 mL, 500 mg, part number 8B-S002-HCH, Waters SepPak C18, 1 to 10g, or equivalent.

6.9.2. Waters Oasis WAX 150 mg/6 cc (PN 186002493) for the cleanup of solids.

6.9.3. Waters Oasis WAX 500 mg/6 cc (PN 186004647) for extraction of PFAS from aqueous sample.

6.9.4. Phenomonex Gemini 3 µm C18 110Å, 50 X 2 mm, Part No. 00B-4439-B0.

6.9.5. Phenomonex Luna 5 µm C18(2) 100Å, 30 X 3 mm, Part No. 00A-4252-Y0.

6.10. Granulated carbon.

6.11. Vacuum manifold for Solid Phase Extraction (SPE).

6.12. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.13. Water bath: Heated with concentric ring cover capable of temperature control (±5°C) up to 95°C. The bath must be used in a fume hood.

6.14. Plastic tub for an ice bath, AKRO-N.S.T. part No. 35-180 or equivalent.

6.15. pH indicator paper, wide range.

6.16. Bottle rotating apparatus for soil extractions.

6.17. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent.

6.18. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – Either of the instruments described below, or equivalent, may be used for this method. Both HPLC are equipped with a refrigerated autosampler, an injection valve, and a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.1 or equivalent.

6.18.1. Waters LC/MS/MS

This consists of a Waters Acquity UPLC system interfaced with a Waters Quattro Premier tandem mass spectrometer. The instrument control and data acquisition software is MassLynx version 4.1, or equivalent.

6.18.1.1. Analytical column: Waters Acquity UPLC BEH C18 1.7 um, 3.0 mm x 150 mm, Part No. 186004690,

6.18.1.2. PFAS Isolator column, Waters Acquity UPLC BEH Shield RP-18, 1.7 um, 2.1 mm x 50 mm, PN 186004476, or equivalent.

This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.18.2. SCIEX LC/MS/MS

This system consists of a Shimadzu HPLC interfaced with a SCIEX 5500 Triple Quad MS. The instrument control and data acquisition software is SCIEX Analyst, version 1.6.3 or equivalent.

6.18.2.1. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

6.18.2.2. Phenomenex Gemini C_{18} 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.

6.18.2.3. PFAS Isolator column, Phenomenex Luna C_{18} 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E0 or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.19. Preventive and routine maintenance is described in the table below

HPLC/MS/MS Preventative Maintenance
<u>As Needed:</u> Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.
<u>Daily (When in use)</u> Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses.

HPLC/MS/MS Preventative Maintenance
Verify that corona and multiplier are functioning.
Semi-Annually Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable.
Annually Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Acetic acid, glacial

7.1.2. Ammonium acetate (20 mM in water)

7.1.3. Ammonium hydroxide (NH4OH), 0.3% in methanol

7.1.4. Hexane

7.1.5. Hydrochloric acid (HCl), 2.0 M solution in water

7.1.6. Hydrochloric acid (HCl), concentrated, reagent grade

7.1.7. Methanol

7.1.8. Potassium hydroxide (KOH), 0.4% in methanol

7.1.9. Potassium persulfate, reagent grade

7.1.10. Ottawa Sand

7.1.11. Sodium hydroxide (NaOH), 0.1N, in water

7.1.12. Sodium hydroxide (NaOH), 10N, reagent grade

7.1.13. Water, Nanopure or Millipore, must be free of interference and target analytes

7.1.14. Methanol-Water, 78:22 vol./vol., prepared by mixing 780 mL methanol and 220 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap.

7.2. Standards

7.2.1. PFAS are purchased as high purity solids (96% or greater) or as certified solutions. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by the manufacturer or vendor.

7.2.1.1. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.15 ng/L or 0.01 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

7.2.2. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at 4 ± 2°C. Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

7.2.3. PFBS, PFHxS, PFHpS, PFOS, PFDS, MPFOS, and many other PFAS are not available in the acid form, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

$Mass_{acid} = \text{Measured } Mass_{salt} \times MW_{acid} / MW_{salt}$

Where: MW_{acid} is the molecular weight of PFAA

MW_{salt} is the molecular weight of the purchased salt.

7.2.4. For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be adjusted by a factor of 1.046.

7.3. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of PFCA and PFSA stock solutions in 80% methanol/water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.4. Initial Calibration (ICAL) Levels (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Perfluoroalkylcarboxylic acids (PFCAs)							
PFBA	0.5	1.0	5.0	20	50	200	400
PFPeA	0.5	1.0	5.0	20	50	200	400
PFHxA	0.5	1.0	5.0	20	50	200	400
PFHpA	0.5	1.0	5.0	20	50	200	400
PFOA	0.5	1.0	5.0	20	50	200	400
PFNA	0.5	1.0	5.0	20	50	200	400
PFDA	0.5	1.0	5.0	20	50	200	400
PFUdA	0.5	1.0	5.0	20	50	200	400
PFDoA	0.5	1.0	5.0	20	50	200	400
PFTrDA	0.5	1.0	5.0	20	50	200	400
PFTeDA	0.5	1.0	5.0	20	50	200	400
PFHxDA	0.5	1.0	5.0	20	50	200	400
PFODA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonic acids (PFSAs)							
PFBS	0.5	1.0	5.0	20	50	200	400
PFHxS *	0.5	1.0	5.0	20	50	200	400
PFHpS	0.5	1.0	5.0	20	50	200	400
PFOS *	0.5	1.0	5.0	20	50	200	400
PFDS	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamides (FOSA)							
FOSA	0.5	1.0	5.0	20	50	200	400
EtFOSA	0.5	1.0	5.0	20	50	200	400
MeFOSA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamidoacetic acids (FOSAA)							
EtFOSAA	0.5	1.0	5.0	20	50	200	400
MeFOSAA	0.5	1.0	5.0	20	50	200	400
Fluorotelomer sulfonates (FTS)							
4:2 FTS	0.5	1.0	2.0	20	50	200	400
6:2 FTS	0.5	1.0	5.0	20	50	200	400

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
8:2 FTS	0.5	1.0	5.0	20	50	200	400
Labeled Isotope Dilution Analytes (IDA)							
13C4-PFBA	50	50	50	50	50	50	50
13C5-PFPeA	50	50	50	50	50	50	50
13C2-PFHxA	50	50	50	50	50	50	50
13C4-PFHpA	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50
13C2-PFDA	50	50	50	50	50	50	50
13C2-PFUdA	50	50	50	50	50	50	50
13C2-PFDoA	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50
13C3-PFBS	50	50	50	50	50	50	50
13C2-PFTeDA	50	50	50	50	50	50	50
13C2-PFHxDA	50	50	50	50	50	50	50
13C8-FOSA	50	50	50	50	50	50	50
d5-EtFOSA	50	50	50	50	50	50	50
d3-MeFOSA	50	50	50	50	50	50	50
d5-EtFOSAA	50	50	50	50	50	50	50
d3-MeFOSAA	50	50	50	50	50	50	50
M2-4:2FTS @	50	50	50	50	50	50	50
M2-6:2FTS	50	50	50	50	50	50	50
M2-8:2FTS	50	50	50	50	50	50	50
Internal Standard (IS)							
13C2-PFOA	50	50	50	50	50	50	50

* Both branched and linear isomers are used.

@ - This compound is used as a reverse surrogate for the TOP analysis.

Note: Sample extracts are in 80% MeOH/H_2O.

Note- The above calibration limits are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.

7.4.1. A technical (qualitative) grade PFOA standard is analyzed initially, then after initial calibration when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.

7.5. Initial Calibration Verification Standard (ICV)

A second source solution for PFAS is purchased from the same vendor; the PFC-MXB contains most of the target analytes in this mixture and is used as an ICV. A few compounds are not available in this mixture, may not be available as another lot, and are not available from another vendor. For these analytes only, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered. The IDA and IS are added at a fixed concentration of 50 ng/mL.

7.6. LCS/Matrix PFC Spike Solution, 20 ng/mL

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 20 ng/mL in methanol.

7.7. PFC Isotope Dilution Analyte Solution, 50 ng/mL

The PFC-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each compound at a concentration of 50 ng/mL in methanol.

7.8. Reverse Surrogate Solution, 1000 ng/mL

The reverse surrogate solution is prepared by diluting M2-4:2 FTS to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all samples for the TOP assay to monitor the efficiency of the oxidation process.

7.9. Internal Standard Solution, 1000 ng/mL

The internal standard solution is prepared by diluting 13C2-PFOA to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all extracts prior to analysis. Non-concentrated extracts are fortified with a 5X dilution of this solution.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Soil samples are collected in pre-cleaned 8 oz. HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6°C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6°C. Water samples must be extracted within 14 days of collection. Soil samples must also be extracted within 14 days of collection. Tissue samples must be extracted within 1 year of collection if stored at -20°C. Extracts must be refrigerated at 0 - 6°C, and analyzed within 40 days from extraction.

NOTE: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted time stability studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6°C. The 14/40 day holding times given above are based on the stability study and general EPA convention for the holding time of extractable organic compounds in water and soil.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.

9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD, an LCSD may be substituted if batch precision is required by the program or client. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.

9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For solid samples, the method blank is an aliquot of Ottawa sand. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, and then implemented when target analytes are detected in the method blank above the reporting limit or when IDA recoveries are outside of the control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.

9.3.1. If the MB produces a peak within the retention time window of any of the analytes, determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.4. Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit/LOQ for each analyte, or less than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. If contamination remains, the contaminated samples should be re-prepared and reanalyzed with a new MB and batch-specific QC samples.

9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples and Ottawa sand for solids) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC, and all associated samples are required if the LCS is deemed unacceptable. See

WS-PQA-0003 for specific acceptance criteria. The control limits for the LCS are stored in TALS.

9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a nonconformance memo, then implemented when recoveries of any spiked analyte are outside of the control limits provided by TALS or by the client.

9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.

9.7. Initial calibration verification (ICV) – When available, a second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.

9.8. Isotope Dilution Analytes

9.8.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

9.8.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-nose ratio is greater than 10:1.

9.8.2.1. Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.

9.8.2.2. Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10%.

9.8.2.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

9.8.2.3. Projects performed under the auspices of the DOD/DOE must meet QSM 5.1 specific criteria for IDA recoveries which are 50-150%. If QC or field samples do not meet this criteria then re-extraction is required.

9.9. Internal Standard

9.9.1. The Internal Standard (IS) is added to each field and QC samples prior to analysis. The IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

9.9.2. Sample IS response (peak area) must be within ± 50% of the response (peak area) in the most recent CCV.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003 "Calibration Curves and Selection of Calibration Points".

10.2. Routine instrument operating conditions are listed in the table in Section 11.16.

10.3. Instrument Tuning

Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound (native and IDA) into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary. The mass assignments must be within ± 0.5 amu of the values shown in the table in Section 11.16.

10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to, new columns or pump seals. A new calibration is not required after minor maintenance.

10.5. With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points.

10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.

10.8. Initial Calibration

10.8.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.

10.8.1.1. A minimum of five analytical standards is used when using average response factor and/or linear calibration fits.

10.8.1.2. A minimum of six analytical standards is used when a quadratic fit is used to generate the curve.

10.8.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

10.8.2.1. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated against an identically labeled analog must be < 35% for the curve to be valid.

10.8.2.2. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated against a closely related labeled analog IDA must be < 50% for the curve to be valid.

10.8.2.3. For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

10.8.2.4. The Internal Standard (IS) response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

10.8.2.5. Projects performed under the auspices of the DOD/DOE must meet QSM 5.1 specific criteria for initial calibration: The %RSD of the RFS for all analytes must be <20%. Linear or non-linear calibrations must have r^2>0.99 for each analyte. Analytes must be within 70-130% of their true value for each calibration standard.

10.9. Calibration Curve Fits

10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-P-003, “Calibration Curves and Selection of Calibration Points”.

10.9.2. The linear curve uses the following function:

Equation 1

$$y = bx + c$$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

x = concentration
b = slope
c = intercept

10.9.3. The quadratic curve uses the following function:

Equation 2

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

10.9.4. Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.

- There should be no carryover at or above 1/2 MRL after a high CAL standard.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.9.5. Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.10. Initial Calibration Blank (ICB)

10.10.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 80:20 methanol:water blank.

10.10.2. The result for the calibration blank must be less than the reporting limit.

10.10.3. If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.10.4. Projects performed under the auspices of the DOD/DOE must meet QSM 5.1 specific criteria for instrument blanks. One is required immediately following the highest standard analyzed and *daily prior to sample analysis*. The instrument blank must be < ½ the LOQ.

10.11. Initial Calibration Verification (ICV)

10.11.1. Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

10.11.2. The recovery for the ICV must meet the appropriate following criteria:

10.11.2.1. The native analyte must be within or equal to 60-140% for all native analytes quantitated against an identically labeled analog IDA.

10.11.2.2. The native analyte must be within or equal to 50-150% for all native analytes quantitated against a closely related labeled analog IDA.

10.11.2.3. The IDA must be within or equal to 50-150%.

10.11.3. Projects performed under the auspices of the DOD/DOE must meet QSM 5.1 specific criteria for the ICV. Analyte concentrations must be within ±30% of their true values for all analytes, IDA and target.

10.11.4. See Section 9.8 for corrective actions in the event that the ICV does not meet the criteria above.

10.12. Continuing Calibration Verification (CCV)

Analyze a CCV at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid-level range of the curve and should vary throughout the run from low level (LOQ/RL) to mid level. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started. In addition, the low standard in the curve must be analyzed and must be within ± 50% of the expected value.

10.12.1. The recovery for the CCV standards must be equal to or within 60-140% for all natives quantitated against an identically labeled analog and equal to or within 50% to 150% for all natives quantitated against a closely related labeled analog. The recovery for the IDA must be within or equal to 50-150%.

10.12.2. The Internal Standard (IS) response (peak area) must be within ± 50% from the response (peak area) from the midpoint of the initial calibration.

10.12.2.1. Sample IS response (peak area) must be within ± 50% of the response (peak area) in the most recent CCV.

10.12.3. If this is not achieved, the instrument has drifted outside the calibration limits. The instrument must be recalibrated.

10.12.4. Projects performed under the auspices of the DOD/DOE must meet QSM 5.1 specific criteria for CCV. All analyte concentrations must be within ± 30% of their true value. Additionally, prior to analysis and at least once every 12 hours an instrument sensitivity check (ISC) must be analyzed. The analyte concentrations must be at LOQ and the concentrations must be within ± 30% of their true value. This can be used as a CCV.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.2. Water Sample Preparation

11.2.1. Visually inspect samples for the presence of settled and/or suspended sediment/particulate. Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.

11.2.2. If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.2.3. Measure 250 mL of each sample using a graduated cylinder and pour into a labeled 16 oz. polyethylene (HDPE) bottle. *Prepare separate aliquots of 1.0 mL if EtFOSA and/or MeFOSA are requested.)*

11.2.3.1. Alternatively, weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume.

11.2.4. Prepare additional aliquots of a field sample for the MS/MSD, if requested.

11.2.5. Prepare two 250 mL aliquots of HPLC-grade water for the method blank and LCS (or 1.0 mL if EtFOSA and/or MeFOSA are requested.)

11.2.6. Spike the LCS and MS/MSD (if requested) with 0.5 mL of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 40 ng/L. If EtFOSA and/or MeFOSA are required, increase the amount of LCS Matrix PFC spike solution added to 2.5 mL.

11.2.7. Add 0.5 mL of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial. If EtFOSA and/or MeFOSA are requested increase the amount of IDA added to 2.5 mL.

11.2.8. If EtFOSA and/or MeFOSA are requested, add 100uL of IS and then adjust the final volume (FV) of these aliquots to 5.0 mL with MeOH. QC samples, LCS, MS, and MSD will require concentration via nitrogen to adjust the FV to 5.0 mL. Vortex each sample. Then, transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.3. Solid Phase Extraction (SPE) of Aqueous Samples

(Do not perform SPE extraction if EtFOSA and/or MeFOSA are requested.)

The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the background check.

11.3.1. Condition the SPE cartridges (Waters WAX, 500 mg/6 cc) by passing the following without drying the column.

NOTE: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.3.2. Wash with 5.0 mL of 0.3% NH_4OH/methanol.

11.3.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200 uL remains on top to keep column wet. After this step, the columns cannot go dry until the completion of loading and rinsing samples.

11.3.4. Appropriately label the columns and add the reservoir to the column.

11.3.5. Add samples to the columns and with vacuum, pull the entire 250 mL aliquot of the sample through the cartridge at a rate of approximately 2 to 5 drops per second.

11.3.6. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.

11.3.7. After the sample and water rinse have completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.

11.4. SPE Column Wash of Aqueous Samples with Hexane

11.4.1. Load the first 5 mL of hexane to soak for five minutes and then elute to waste.

11.4.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.4.3. Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried before continuing.

11.5. SPE Elution of Aqueous Samples – using 15 mL polypropylene test tubes as receiving tubes in the SPE manifold.

11.5.1. Rinse sample bottles with 5 mL of 0.3% NH_4OH/methanol and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.5.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of 0.3% NH_4OH/methanol. The total collection should be approximately 10 mL.

11.5.3. Note: If the extracts will not be concentrated elute extract with a total of 8 mL of 0.3% NH_4OH/methanol.

11.6. Extract Concentration for Aqueous Extracts (Note, if the extract will not be concentrated, proceed to Section 11.7.)

11.6.1. Prior to concentrating each sample, add 100 uL of water.

11.6.2. Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 100 uL of water remains.

11.6.2.1. This blow down must take a minimum of 3.5 hours.

11.6.2.2. Extracts can not remain in the water bath longer than 5 minutes once concentrated.

11.6.3. Add 300 uL of the 78:22 methanol:water solution and mix the contents well using a vortex mixer.

11.6.4. Add 100 uL of Internal Standard (IS) solution to each extract and vortex to mix.

11.6.5. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.6.6. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.6.7. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.7. Final volume for non-concentrated extract

11.7.1. If the extract does not undergo concentration add 0.5 mL of IS and 2 mL of water to the extract. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.7.2. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.7.3. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps cannot be used due to detection of low level concentration of PFAS.

11.8. Soil, Sediment and Tissue Sample Preparation and Extraction

11.8.1. Visually inspect soil samples for homogeneity.

11.8.2. Weigh a representative 5 g aliquot of soil, sediment or 1 g of tissue sample into a 50 mL HDPE wide-mouth bottle. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested. *(Prepare separate aliquots if EtFOSA and/or MeFOSA are requested.)*

11.8.3. For the method blank and LCS matrix, use 5 g each of Ottawa sand or 0.1 g of oil.

11.8.4. Spike the LCS and MS/MSD (if requested) with 1.0 mL of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 4.0 ng/g.

11.8.5. Add 1.0 mL of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.8.6. Cap the bottles and allow the spike to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.

11.8.7. Add 20 mL of 0.4% KOH/methanol to each sample.

11.8.8. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.8.9. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.

11.8.10. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.8.11. Collect and decant the KOH/methanol extract to a new 50 mL centrifuge tube.

11.8.12. Add another 2 mL of 0.4% KOH/methanol solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.8.13. Combine the rinsate to the first corresponding tubes.

11.8.14. To the final KOH/methanol extract, add 2 mL of water to each. *(Omit this step if EtFOSA and/or MeFOSA are requested.)*

11.8.15. Concentrate the KOH/methanol/water extract under nitrogen to less than 2 mL, and dilute with water to 15 mL final volume. *(Omit this step if EtFOSA and/or MeFOSA are requested.)*

11.8.16. Acidify with 80 uL of glacial acetic acid, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 to 8.

11.8.17. Centrifuge at 3500 rpm for 15 minutes.

11.9. Solid Extract Cleanup by SPE

(Do not perform SPE clean up if EtFOSA and/or MeFOSA are requested. Proceed directly to Section 11.11)

11.9.1. Set up WAX 150 mg/6 cc SPE columns for sample cleanup using vacuum manifold.

11.9.2. Condition the SPE cartridges by passing the following without drying the column.

NOTE: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.9.3. Wash with 5.0 mL of 0.3% NH_4OH/methanol.

11.9.4. Wash with 10 mL of 0.1N NaOH/water. Close valve when ~ 500uL remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

11.9.5. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.

11.9.6. Rinse the sample tube with 5 mL of water and add to the SPE column.

11.9.7. Dry the columns with vacuum for 15 minutes.

11.10. SPE Column Wash of Solid Extracts with Hexane

11.10.1. Load the first 5 mL of hexane to soak for five minutes, and elute to waste.

11.10.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.10.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.11. SPE Elution of Solid Extracts – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

11.11.1. Rinse extraction bottles with 5 mL of 0.3% NH_4OH/methanol and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.11.2. Repeat extract bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of 0.3% NH_4OH/methanol. The total collection should be approximately 10 mL.

11.11.3. Note: If the extracts will not be concentrated elute extract with a total of 8 mL of 0.3% NH_4OH/methanol.

11.12. Extract Concentration for Solid Samples (Note, if the extract will not be concentrated, proceed to Section 11.7)

11.12.1. Prior to concentrating each sample, add 200 uL of water.

11.12.2. Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 200 uL of water remains.

11.12.2.1. This blow down must take a minimum of 3.5 hours.

11.12.2.2. Extracts can not remain in the water bath longer than 5 minutes once concentrated.

11.12.2.3. Add 600 uL of the 78:22 methanol:water solution and mix the contents well using a vortex mixer.

11.12.2.4. Add 200 uL of Internal Standard (IS) solution to each extract and vortex to mix.

11.12.3. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.12.4. Seal the vial with a polypropylene screw cap. *Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.*

11.13. Product/Dispersion Samples

11.13.1. Check the solubility of the material in both methanol and water

11.13.1.1. If the material is soluble in water, dilute 0.5 mL of sample into 250 mL of DI water and proceed to Section 11.3 (follow water extraction procedures). Fortify sample appropriately with IDA or PFC spike solution, see Section 11.2.

11.13.1.2. If the material is soluble in methanol, dilute 1 g (if solid) or 1 mL (if liquid) of material into 10 mL of methanol (MeOH).

11.13.1.2.1. If the material does not completely dissolve, contact your immediate supervisor.

11.13.2. Take 100 uL of the 10 mL solution and dilute it to 10 mL in MeOH.

11.13.3. Take a 1 mL aliquot of this solution (effective dilution of 1000x (1 mg for solid or 0.001 mL for liquid)) and fortify with 0.5 mL of labeled IDA solution (Section 7.7).

11.13.4. DO NOT PASS EXTRACT THROUGH SPE CARTIRIDGE (omit steps 11.9 – 11.11).

11.13.5. Proceed to Section 11.6 of this SOP for extract concentration.

11.14. TOP (Total Oxidizable Precursor) Assay

11.14.1. Prepare 3-250 mL HDPE containers with HPLC grade water to create the needed QC Samples (MB, LCS/LCSD).

11.14.2. Prepare enough 125 mL HDPE containers as needed for all “Pre” and “Post” samples, including QC. Label each appropriately.

11.14.3. Spike the “Pre” and “Post” MB 125 mL containers with 25 uL of the reverse surrogate solution of M2-4:2 FTS (Section 7.8).

11.14.4. Spike the “Pre” and “Post” LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution (Section 7.6), both regular and “add-on”, and 25 uL of the reverse surrogate solution (Section 7.8).

11.14.5. Remove the methanol solvent from all Post QC sample 125 mL containers (MB and LCS/LCSD) by using N2 evaporation.

11.14.6. Subsample 100 mL aliquots of water from each field sample and QC from the 250 mL containers into each of the corresponding 125 mL containers for both the “Pre” and “Post” samples.

11.14.7. Set aside all “Pre” sample containers.

11.14.8. Add 2g of potassium persulfate and 1.9 mL of 10N NaOH to each “Post” sample container.

11.14.9. Heat each "Post" sample container in a water bath (KD) at 85°C for 6 hours.

11.14.10. After digestion for 6 hours, place the "Post" sample containers in an ice bath for 30 minutes.

11.14.11. Adjust the pH of "Post" samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.14.12. Spike both "Pre" and "Post" samples and their associated QC samples with 0.5 mL of PFC IDA solution (Section 7.7), both regular and add-on.

11.14.13. Use the following SPE procedure for both "Pre" and "Post" samples:

11.14.13.1. Set up WAX 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.14.13.2. Establish a sample loading flow rate of 1 mL/minute for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.14.13.3. Wash/condition the SPE column with 5 mL of 0.3% NH_4OH/Methanol, then 5 mL water.

11.14.13.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 1 mL/minute.

11.14.13.5. Add 5 mL rinse water

11.14.13.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.14.13.7. Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.14.13.8. Allow the column to dry well using vacuum with a flow rate of 1 mL/minute for 5 minutes. Columns must be dry before continuing.

11.14.13.9. Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 5 mL of 0.3% NH_4OH/methanol, and add to the SPE cartridge as eluent.

11.14.13.10. Repeat with another 5 mL of 0.3% NH_4OH/methanol.

11.14.13.11. Collect the 10 mL of eluent and concentrate per Section 11.6.

11.15. Other Types of Sample Cleanup

11.15.1. Freezing technique to remove lipids.

If samples contain lipids then freeze the methanolic extract and QC extracts at -20°C for at least 1 hour. Collect the solvent layer.

11.15.2. Cleanup with graphitized carbon which may also be used to remove organic interferences.

11.15.2.1. Add 100 mg of graphitized carbon to each sample extract and QC extracts.

11.15.2.2. Shake vigorously and then let sit for 10 minutes.

11.15.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.

11.15.2.4. Decant the solvent layer

11.15.3. Concentrate each sample under a gentle stream of nitrogen to approximately 0.5 mL.

11.15.4. Add 200 uL of Millipore water to each sample.

11.15.5. Bring the final volume to 1.0 mL with methanol (80% methanol/20% water).

11.15.6. Filter through a 0.45 µm syringe filter as necessary or centrifuge the extracts to obtain a clear supernant. *Note: Syringe filter should be checked for PFAS background before using.*

WARNING: Application of excessive pressure has caused disc filters to rupture and burst. Exercise discretion when filtering.

11.16. Instrument Analysis

Suggested operating conditions are listed below for the Waters LCMS system:

Recommended Instrument Operating Conditions					
HPLC Conditions (Waters Acquity UPLC)					
Column (Column temp = 50°C)	Waters Acquity BEH 1.7µm C18, 3.0 x 150 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	**Time**	**%A**	**%B**	**Curve**	**Flow Rate mL/min.**
	0	98	2	6	0.30

	1	98	2	6	0.30
	2	50	50	6	0.30
	12	10	90	6	0.30
	12.5	0	100	6	0.30
	16	0	100	6	0.30
	16.2	98	2	6	0.30
	Maximum pressure limit = 15,000 psi				
Injection Size	10 µL (fixed amount throughout the sequence)				
Run Time	~20 minutes				
Mass Spectrometer Interface Settings (Quattro Premier XE)					
MS Interface Mode	ESI Negative Ion				
Capillary (kV)	2.8				
Cone (V)	Varies from 8.0 to 65				
Extractor (V)	3				
Source Temp	135ºC				
Desolvation Temp	350ºC				
Cone Gas (nitrogen) Flow	25 L/hour				
Desolvation Gas (nitrogen) Flow	1100 L/hour				

Recommended Instrument Operating Conditions						
Mass Spectrometer Scan Settings (Quattro Premier XE)						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
PFBA	Native analyte	213 > 169	0.02	8	10	1
13C4-PFBA	IDA	217 > 172	0.02	12	10	1
PFPeA	Native analyte	263 > 219	0.02	10	10	2
13C5-PFPeA	IDA	268 > 223	0.02	11	9	2
PFBS	Native analyte	299 > 80	0.02	45	35	2
PFBS_2	Native analyte	299 > 99	0.02	45	35	2
13C3-PFBS	IDA	302 > 83	0.02	45	35	2
PFHxA	Native analyte	313 > 269	0.02	10	10	3
PFHxA_2	Native analyte	313 > 119	0.02	10	10	3
13C2-PFHxA	IDA	315 > 270	0.02	12	9	3
PFHpA	Native analyte	363 > 319	0.02	10	10	4
PFHpA_2	Native analyte	363 > 169	0.02	10	10	4
13C4-PFHpA	IDA	367 > 322	0.02	12	10	4
PFHxS	Native analyte	399 > 80	0.02	55	35	4
PFHxS_2	Native analyte	339 > 99	0.02	55	35	4
18O2-PFHxS	IDA	403 > 84	0.02	50	40	4

PFOA	Native analyte	413 > 369	0.02	12	10	5
PFOA_2	Native analyte	413 > 169	0.02	12	10	5
13C2-PFOA	IS	415 > 370	0.02	12	12	5
13C4-PFOA	IDA	417 > 372	0.02	12	12	5
PFHpS	Native analyte	449 > 80	0.02	60	38	5
PFHpS_2	Native analyte	449 > 99	0.02	60	38	5
PFNA	Native analyte	463 > 419	0.02	16	10	7
PFNA_2	Native analyte	463 > 169	0.02	16	10	7
13C5-PFNA	IDA	468 > 423	0.02	12	12	7
PFOS	Native analyte	499 > 80	0.02	60	40	6
PFOS_2	Native analyte	499 > 99	0.02	60	40	6
13C4-PFOS	IDA	503 > 80	0.02	35	48	6
PFDA	Native analyte	513 > 469	0.02	16	12	8
PFDA_2	Native analyte	513 > 169	0.02	16	12	8
13C2-PFDA	IDA	515 > 470	0.02	14	12	8
PFUdA	Native analyte	563 > 519	0.02	15	12	10
PFUdA_2	Native analyte	563 > 169	0.02	15	12	10
13C2-PFUdA	IDA	565 > 520	0.02	14	12	10
PFDS	Native analyte	599 > 80	0.02	74	48	10
PFDS_2	Native analyte	559 > 99	0.02	74	48	10
FOSA	Native analyte	498 > 78	0.02	40	32	9
13C8-FOSA	IDA	506 > 78	0.02	48	32	9
PFDoA	Native analyte	613 > 569	0.02	15	14	11
PFDoA_2	Native analyte	613 > 169	0.02	15	14	11
13C2-PFDoA	IDA	615 > 570	0.02	16	12	11
PFTrDA	Native analyte	663 > 619	0.02	12	12	11
PFTrDA_2	Native analyte	663 > 169	0.02	12	12	11
PFTeDA	Native analyte	713 > 169	0.02	12	18	11
PFTeDA_2	Native analyte	713 > 219	0.02	12	18	11
13C2-PFTeDA	IDA	715 > 670	0.02	15	15	11
PFHxDA	Native analyte	813 > 769	0.02	18	15	12
PFHxDA_2	Native analyte	813 > 169	0.02	18	15	12
PFODA	Native analyte	913 > 869	0.02	20	16	12
PFODA_2	Native analyte	913 > 169	0.02	20	16	12
13C2-PFHxDA	IDA	815 > 770	0.02	18	15	12
EtFOSA	Native analyte	526 > 169	0.02	45	36	11
d5-EtFOSA	IDA	531 >169	0.02	40	30	11
MeFOSA	Native analyte	512 > 169	0.02	45	25	11
d5-MeFOSA	IDA	515 > 169	0.02	40	30	11
EtFOSAA	Native analyte	584 > 419	0.02	35	20	9

d5-EtFOSAA	IDA	589 > 419	0.02	30	25	9
MeFOSAA	Native analyte	570 > 419	0.02	30	28	9
d3-MeFOSAA	IDA	573 > 419	0.02	30	25	9
4:2FTS	Native analyte	327 > 307	0.02	40	30	5
M2-4:2FTS	Reverse Surrogate	329 . 309	0.02	40	30	5
6:2FTS	Native analyte	427 > 407	0.02	40	30	5
M2-6:2FTS	IDA	429 > 409	0.02	40	28	5
8:2FTS	Native analyte	527 > 507	0.02	40	28	8
M2-8:2FTS	IDA	529 > 509	0.02	40	28	8

Recommended Instrument Operating Conditions				
Retention Times & Quantitation (Quattro Premier XE)				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	4.77	13C4-PFBA	4.79	Isotope Dilution
PFPeA	5.90	13C5-PFPeA	5.92	Isotope Dilution
PFBS	6.01	13C3-PFBS	6.01	Isotope Dilution
PFHxA	7.22	13C2-PFHxA	7.25	Isotope Dilution
PFHpA	8.57	13C4-PFHpA	8.59	Isotope Dilution
PFHxS	8.60	18O2-PFHxS	8.64	Isotope Dilution
PFOA	9.80	13C4-PFOA	9.83	Isotope Dilution
PFHpS	9.80	13C4-PFOS	10.90	Isotope Dilution
PFNA	10.88	13C5-PFNA	10.92	Isotope Dilution
PFOS	10.87	13C4-PFOS	10.90	Isotope Dilution
PFDA	11.82	13C2-PFDA	11.86	Isotope Dilution
FOSA	12.41	13C8-FOSA	12.46	Isotope Dilution
PFDS	12.57	13C4-PFOS	10.90	Isotope Dilution
PFUdA	12.62	13C2-PFUdA	12.66	Isotope Dilution
PFDoA	13.32	13C2-PFDoA	13.34	Isotope Dilution
PFTrDA	13.91	13C2-PFDoA	13.34	Isotope Dilution
PFTeDA	14.39	13C2-PFTeDA	14.39	Isotope Dilution
PFHxDA	15.16	13C2-PFHxDA	15.16	Isotope Dilution
PFODA	15.57	13C2-PFHxDA	15.16	Isotope Dilution
EtFOSA	14.13	d-EtFOSA	14.11	Isotope Dilution
MeFOSA	13.73	d-MeFOSA	13.73	Isotope Dilution
EtFOSAA	12.63	d5-EtFOSAA	12.62	Isotope Dilution
MeFOSAA	12.3	d3-MeFOSAA	12.28	Isotope Dilution
4:2FTS	7.02	M2-6:2FTS	10.08	Isotope Dilution
6:2FTS	10.08	M2-6:2FTS	10.08	Isotope Dilution
8:2FTS	11.95	M2-8:2FTS	11.95	Isotope Dilution

Recommended Instrument Operating Conditions				
HPLC Conditions (Shimadzu HPLC)				
Column (Column temp = 45°C)	Phenomenex Gemini 3 µm C18 110Å, 50 X 2 mm			
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol			
Gradient Program	**Time**	**%A**	**%B**	**Flow Rate mL/min.**
	0	90	10	0.60
	0.1	45	55	0.60
	4.5	1	99	0.60
	4.95	1	99	0.60
	5	90	10	0.60
	Maximum pressure limit = 5,000 psi			
Injection Size	2 µL (fixed amount throughout the sequence). If non-concentrated extract then use 20 uL.			
Run Time	~6.6 minutes			

Mass Spectrometer Interface Settings (SCIEX 5500)	
MS Interface Mode	ESI Negative Ion
Ion Spray Voltage (kV)	4.5
Entrance Potential (V)	5
Declustering Potential (V)	25
Desolvation Temp	600ºC
Curtain Gas	35 psi
Collision Gas	8 psi

Recommended Instrument Operating Conditions
Mass Spectrometer Scan Settings (SCIEX 5500)

Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFBA	Native analyte	212.9 > 169	0.011	-5	-12	-25	-31	1.74
13C4-PFBA	IDA	217 > 172	0.011	-5	-12	-25	-31	1.74
PFBS	Native analyte	298.9 > 80	0.011	-6	-58	-55	-37	1.76
PFBS_2	Native analyte	298.9 > 99	0.011	-5	-40	-55	-12	1.76
13C3-PFBS	IDA	301.9 > 83	0.011	-5	-40	-55	-12	1.76
PFPeA	Native analyte	262.9 > 219	0.011	-7	-12	-20	-34	1.99
13C5-PFPeA	IDA	267.9 > 223	0.011	-7	-12	-20	-35	1.99
4:2 FTS	Native Analyte	327 > 307	0.011	-7	-32	-50	-10	2.10
M2-4:2FTS	Reverse Surrogate	329 > 309	0.011	-7	-32	-50	-10	2.10

Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings (SCIEX 5500)								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFHxA	Native analyte	313 > 269	0.011	-5	-12	-25	-37	2.25
PFHxA_2	Native analyte	313 > 119	0.011	-5	-12	-25	-37	2.25
13C2-PFHxA	IDA	315 > 270	0.011	-5	-12	-25	-38	2.25
PFHpA	Native analyte	363 > 319	0.011	-6	-12	-25	-41	2.57
PFHpA_2	Native analyte	363 > 169	0.011	-6	-12	-25	-41	2.57
13C4-PFHpA	IDA	367 > 322	0.011	-6	-12	-25	-41	2.57
PFHxS	Native analyte	399 > 80	0.011	-12	-74	-60	-43	2.59
PFHxS_2	Native analyte	399 > 99	0.011	-12	-74	-60	-43	2.59
18O2-PFHxS	IDA	403 > 84	0.011	-12	-74	-60	-43	2.59
6:2 FTS	Native analyte	427 > 407	0.011	-7	-32	-50	-10	2.91
M2-6:2FTS	IDA	429 > 409	0.011	-7	-32	-50	-10	2.91
PFOA	Native analyte	413 > 369	0.011	-6	-14	-25	-44	2.93
PFOA_2	Native analyte	413 > 169	0.011	-5	-22	-25	-12	2.93
13C4-PFOA	IDA	417 > 372	0.011	-6	-14	-25	-44	2.93
13C2-PFOA	IS	415 > 370	0.011	-6	-14	-25	-44	2.93
PFHpS	Native analyte	449 > 80	0.011	-11	-88	-65	-46	2.94
PFHpS_2	Native analyte	449 > 99	0.011	-11	-88	-65	-46	2.94
PFNA	Native analyte	463 > 419	0.011	-6	-14	-25	-47	3.29
PFNA_2	Native analyte	463 > 169	0.011	-6	-14	-25	-47	3.29
13C5-PFNA	IDA	468 > 423	0.011	-6	-14	-25	-48	3.29
PFOS	Native analyte	499 > 80	0.011	-9	-108	-65	-50	3.29
PFOS_2	Native analyte	499 > 99	0.011	-5	-58	-65	-12	3.29
13C4-PFOS	IDA	503 > 80	0.011	-9	-108	-65	-50	3.29
PFDA	Native analyte	513 > 469	0.011	-6	-16	-25	-51	3.65
PFDA_2	Native analyte	513 > 169	0.011	-6	-16	-25	-51	3.65
13C2-PFDA	IDA	515 > 470	0.011	-6	-16	-25	-51	3.65
8:2 FTS	Native analyte	527 > 507	0.011	-7	-40	-50	-15	3.65
M2-8:2FTS	IDA	529 > 509	0.011	-7	-40	-50	-15	3.65
PFOSA	Native analyte	498 > 78	0.011	-8	-85	-60	-50	3.7
13C8-PFOSA	IDA	506 > 78	0.011	-8	-85	-60	-50	3.7
N-MeFOSAA	Native analyte	570 > 419	0.011	-7	-36	-40	-15	3.82
d3-MeFOSAA	IDA	573 > 419	0.011	-7	-36	-40	-15	3.82
PFDS	Native analyte	599 > 80	0.011	-11	-118	-85	-54	3.96
PFDS_2	Native analyte	599 > 99	0.011	-11	-118	-85	-54	3.96
PFUdA	Native analyte	563 > 519	0.011	-7	-18	-25	-54	3.97
PFUdA_2	Native analyte	563 > 169	0.011	-7	-18	-25	-54	3.97
13C2-PFUdA	IDA	565 > 520	0.011	-7	-18	-25	-54	3.97
N-EtFOSAA	Native analyte	584 > 419	0.011	-7	-36	-50	-15	3.99
d5-EtFOSAA	IDA	589 > 419	0.011	-7	-36	-50	-15	3.99
MeFOSA	Native analyte	512 > 169	0.011	-7	-37	-75	-15	4.21
d3-MeFOSA	IDA	515 > 169	0.011	-7	-37	-75	-15	4.21

Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings (SCIEX 5500)								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFDoA	Native analyte	613 > 569	0.011	-5	-18	-25	-54	4.3
PFDoA_2	Native analyte	613 > 169	0.011	-5	-18	-25	-54	4.3
13C2-PFDoA	IDA	615 > 570	0.011	-5	-18	-25	-54	4.3
EtFOSA	Native analyte	526 > 169	0.011	-7	-37	-75	-15	4.39
d5-EtFOSA	IDA	531 > 169	0.011	-7	-37	-75	-15	4.39
PFTrDA	Native analyte	663 > 619	0.011	-7	-20	-25	-54	4.56
PFTrDA_2	Native analyte	663 > 169	0.011	-7	-20	-25	-54	4.56
PFTeDA	Native analyte	713 > 169	0.011	-2	-22	-25	-10	4.79
PFTeDA_2	Native analyte	713 > 219	0.011	-7	-36	-25	-30	4.79
13C2-PFTeDA	IDA	715 > 670	0.011	-2	-22	-25	-10	4.79
PFHxDA	Native analyte	813 > 769	0.011	-7	-24	-25	-54	5.25
PFHxDA_2	Native analyte	813 > 169	0.011	-7	-24	-25	-54	5.25
13C2-PFHxDA	IDA	815 > 770	0.011	-7	-24	-25	-54	5.25
PFODA	Native analyte	913 > 869	0.011	-7	-26	-25	-54	5.55
PFODA_2	Native analyte	913 > 169	0.011	-7	-26	-25	-54	5.55

Retention Times & Quantitation (SCIEX 550)0)				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	1.54	13C4-PFBA	1.54	Isotope Dilution
PFPeA	1.56	13C5-PFPeA	1.56	Isotope Dilution
PFBS	1.78	13C3-PFBS	1.78	Isotope Dilution
PFHxA	2.03	13C2-PFHxA	2.03	Isotope Dilution
PFHpA	2.36	13C4-PFHpA	2.36	Isotope Dilution
PFHxS	2.37	18O2-PFHxS	2.37	Isotope Dilution
PFOA	2.71	13C4-PFOA	2.71	Isotope Dilution
PFHpS	2.72	13C4-PFOS	3.09	Isotope Dilution
PFNA	3.09	13C5-PFNA	3.09	Isotope Dilution
PFOS	3.09	13C4-PFOS	3.09	Isotope Dilution
PFDA	3.45	13C2-PFDA	3.45	Isotope Dilution
FOSA	3.43	13C8-FOSA	3.43	Isotope Dilution
PFDS	3.77	13C4-PFOS	3.09	Isotope Dilution
PFUdA	3.78	13C2-PFUdA	3.78	Isotope Dilution
PFDoA	4.07	13C2-PFDoA	4.07	Isotope Dilution
PFTrDA	4.34	13C2-PFDoA	4.07	Isotope Dilution
PFTeDA	4.58	13C2-PFTeDA	4.58	Isotope Dilution
PFHxDA	4.99	13C2-PFHxDA	4.99	Isotope Dilution
PFODA	5.34	13C2-PFHxDA	4.99	Isotope Dilution

EtFOSA	4.12	d5-EtFOSA	4.12	Isotope Dilution
MeFOSA	3.94	d5-MeFOSA	3.93	Isotope Dilution
EtFOSAA	3.78	d5-EtFOSAA	3.78	Isotope Dilution
MeFOSAA	3.61	d3-MeFOSAA	3.60	Isotope Dilution
4:2 FTS	1.98	M2-6:2 FTS	2.69	Isotope Dilution
6:2FTS	2.69	M2-6:2FTS	2.69	Isotope Dilution
8:2FTS	3.44	M2-8:2FTS	3.44	Isotope Dilution

11.16.1. Tune and calibrate the instrument as described in Section 10.

11.16.2. A typical run sequence is as follows:

- Primer (A number of primers are injected for conditioning of the instrument before analysis, especially when the instrument was idled or changed from a different analysis).
- Blank
- Calibration Curve
- ICB
- ICV
- MB
- LCS
- LCSD (if applicable)
- Sample 1
- Sample 1 MS (if applicable)
- Sample 1 MSD (if applicable)
- Sample 2 (up to sample 10 before next CCV)
- CCV
- Up to 10 samples.
- End sequence with CCV

12. CALCULATIONS

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. It may be necessary to dilute samples due to matrix.

12.2. Qualitative Identification

12.2.1. The retention times of PFAS with labeled standards must be the same as that of the labeled IDA's to within 0.05 min. For PFAS with no labeled standards, the RT must be within ± 0.3 minutes of the ICV and CCV standards. *Note: The IDA RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.3. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.4. Extract concentrations are calculated as below. The first equation applies to the linear fit, the second to the quadratic line fit.

Equation 3

$$\text{Concentration, ng/mL} = \frac{y - c}{b}$$

Equation 4

$$\text{Concentration, ng/mL} = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

x = concentration
a = curvature
b = slope
c = intercept

12.5. Water Sample Result Calculation:

Equation 5

$$\text{Concentration, ng/L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
V_o = Volume of water extracted (L)

12.6. Soil Sample Result Calculation:

Equation 6

$$\text{Concentration, } ng/g = \frac{C_{ex} V_t}{W_s D}$$

Where ng/g = µg/kg and:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
W_s = Weight of sample extracted (g)
D = Fraction of dry solids, which is calculated as follows:

$$\frac{100 - \%\ \text{moisture in sample}}{100} \quad \text{(for dry weight result)}$$

12.7. IDA Recovery Calculation:

Equation 7

$$\%\ Recovery = \frac{A_t Q_{is}}{A_{is} Q_t RRF_{IDA}} X100$$

Where ng/g = µg/kg and:

RF_{IDA} = Response Factor for IDA compound
A_t = Area response for IDA compound
A_{IS} = Area Response for IS compound
Q_{IS} = Amount of IS added
Q_t = Amount of IDA added

12.8. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006 and policy WS-PQA-003. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration of Capability (IDOC)

Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

13.4. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy WS-PQA-003.

14. POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

14.2. Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid-liquid extraction.

14.3. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out:

15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the hazardous waste – landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted solids contaminated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the incineration steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to no less than six inches of the top, or after no more than 75 days, move the steel flammable solvent drum to the waste collection area for shipment.

15.4. Mixed water/methanol waste from soil extraction. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full, to no less than six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.5. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.6. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES

16.1. Cheryl Moody, Wai Chi Kwan, Johnathan W. Martin, Derek C. G. Muir, Scott A. Mabury, “Determination of Perfluorinated Surfactants in Surface Water Samples by Two Independent Analytical Techniques: Liquid Chromatography/Tandem Mass Spectrometry and 19FNMR,” Analytical Chemistry 2001, 73, 2200-2206.

16.2. John Giesy et al., “Accumulation of Perfluorooctane Sulfonate in Marine Mammals”, Environmental Science & Technology, 2001 Vol. 35, No. 8, pages 1593-1598.

16.3. U.S. EPA, “Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method”, EPA 712-C-95-174, August 1995.

16.4. STL Denver White Paper DEN-W-LC-002, "Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, September 5, 2003.

16.5. STL Denver White Paper DEN-W-LC-003, "Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.

16.6. STL Denver White Paper DEN-W-LC-004, "Method Validation Study for Analysis of Perfluorooctanoic Acid in Waters by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, January 26, 2005.

16.7. Waters application note; "Acquity UPLC System for Quantifying Trace Levels of Perfluorinated Compounds with an Acquity PFC Analysis Kit", Peter J. Lee, Evan T. Bernier, Gordon T. Fujimoto, Jeremy Shia, Michael S. Young, and Alice J. Di Gloia, Waters Corporation, Milford, MA. USA.

16.8. US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometery (LC/MS/MS)", Version 1.1, September 2009, J.A. Shoemaker, P.E. Grimmett, B.K. Boutin, EPA Document #: EPA/600/R-08/092

16.9. Erika F. Houtz and David L. Sedlak, "Oxidative Conversion as a Means of Detecting Precursors to Perfluoroalkyl Acids in Urban Runoff," Environmental Science and Technology 46, no. 17 (2012): 9342-49.

17. METHOD MODIFICATIONS

17.1. Modifications from Method 537 are detailed below:

17.1.1. Water sample containers are not preserved with Trizma.

17.1.2. The method has been modified to address soil/solid matrices. The extraction holding time is set at 14 days.

17.1.3. The analyte list has been expanded. The number of labeled analytes has been expanded as well to improve quantitation.

17.1.4. The reporting limits differ as they are all set at one consistent value.

17.1.5. Calibration levels differ from the referenced method.

17.1.6. More labeled analytes are fortified into the samples prior to the extraction process. Most target analytes are quantitated against a labeled analyte.

17.1.7. There is no symmetry requirement.

17.1.8. Calibration, both initial and continuing, has different acceptance criteria due to the longer list of analytes, and the use of isotope dilution quantitation.

17.1.9. The eluents and HPLC configuration differs. As a result the final extract is in 80:20 methanol:water.

17.1.10. The LCS and MS/MSD are spiked at one concentration and do not rotate between a low to high levels.

17.1.11. Samples are not checked for residual chlorine or pH.

17.1.12. A different SPE cartridge (Waters OASIS WAX) is used for the extraction process. As a result solvents and elution procedures are different.

18. ATTACHMENTS

18.1. Attachment 1 - Analysis of Perfluorinated Compounds (PFAS) in Water via In Line Solid Phase Extraction (SPE).

19. REVISION HISTORY

Revisions to Attachment 1 are documented in the attachment.

Revisions prior to 04/10/2017 have been removed and are available in previous versions of this SOP.

19.1. WS-LC-0025, Revision 2.7, Effective 09/20/2017

19.1.1. Section 1.1 table, added 1H,1H,2H,2H-perfluorohexane sulfonate *(4:2).*

19.1.2. Section 1.1, removed “Sample results for PFOA may also be reported as APFO, at the request of the client. (See Section 12.7).”

19.1.3. Section 1.2 and 11.8.2, updated tissue extracted mass and RL.

19.1.4. Section 2.5, removed “and assumes a proportional relationship between the initial calibration and the analyte in the extract. The ratio of the peak response to mass or concentration injected is used to prepare a calibration curve."

19.1.5. Added Section 6.6, "Extract concentrator or nitrogen manifold with water bath heating to 50-55°C".

19.1.6. Added Section 7.1.14, "Methanol-Water, 78:22 vol./vol., prepared by mixing 780 mL methanol and 220 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap."

19.1.7. Section 7.2.1.1, revised "roughly 0.15 pg/L" to "roughly 0.15 ng/L".

19.1.8. Section 7.4 table, added:

4:2 FTS	0.5	1.0	2.0	20	50	200	400

19.1.9. Section 7.4 table, revised Labeled Isotope Dilution Analytes (IDA) Section.

19.1.10. Section 7.4 table, added:

Internal Standard (IS)							
13C2-PFOA	50	50	50	50	50	50	50

19.1.11. Section 7.4, removed "FOSAA may be added to the mix and are added at the same concentration as FOSA."

19.1.12. Added Section 7.9, "Internal Standard Solution, 1000 ng/mL. The internal standard solution is prepared by diluting 13C2-PFOA to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all extracts prior to analysis. Non-concentrated extracts are fortified with a 5X dilution of this solution."

19.1.13. Section 8.1, changed "250 mL" to "8 oz."

19.1.14. Added Sections 9.3.6, 9.8.2.3, 10.10.4, 10.8.2.5, 10.11.3, and 10.12.4 to address DOD QSM 5.1 Table B-15 criteria.

19.1.15. Added Section 9.9, "Internal Standard."

19.1.16. Updated all tables to indicate target analyte quantitation via isotope dilution. Internal standard quantitation is only used to quantitate the IDA recoveries.

19.1.17. Added Section 10.8.2.4, 10.12.2, and 10.12.2.1 to incorporate IS criteria into calibrations.

19.1.18. Section 11.2.1, "Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias."

19.1.19. Added Section 11.2.3.1, "Alternatively, weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume."

19.1.20. Added Section 11.5.3, "Note: If the extracts will not be concentrated elute extract with a total of 8 mL of 0.3% NH_4OH/methanol."

19.1.21. Added Section 11.6.2.3, "Add 300 uL of the 78:22 methanol:water solution and mix the contents well using a vortex mixer."

19.1.22. Added Section 11.6.2.4, "Add 100 uL of Internal Standard (IS) solution to each extract and vortex to mix."

19.1.23. Added Section 11.7, "Final volume for non-concentrated extract".

19.1.24. Revised Section 11.11, "SPE Elution of Solid Extracts".

19.1.25. Revised Section 11.12, "Extract Concentration for Solid Samples".

19.1.26. Removed Section 12.8, "If results are to be reported as ammonium perfluorooctanoate (APFO), instead of PFOA, apply a multiplier of 1.0406 to the sample results to correct for the molecular weight differences between PFOA and APFO or this adjustment can be made during the preparation of the standards used for calibration. (Use one, not both.)"

19.1.27. Removed Section 13.4 – it was a copy of Section 13.2.

19.1.28. Various revisions to fulfill requirements based on DOD/DOE QSM 5.1.

19.1.29. Editorial changes.

19.2. WS-LC-0025, Revision 2.6, Effective 08/15/2017

19.2.1. Section 7.4, added MPFBS, MPFTeDA, and MPFHxDA to the table.

19.2.2. Section 11.15, added 13C-PFBS to the Recommended Instrument Operating Conditions table for SCIEX 5500.

19.2.3. Section 11.15 Recommended Instrument Operating Conditions table, changed the mass transitions for native PFTeDA from 713 > 669 (quant) and 713 > 169 (qualifier) to 713 > 169 (quant) and 713 > 219 (qualifier).

19.2.4. Editorial changes.

19.3. WS-LC-0025, Revision 2.5, Effective 07/10/2017

19.3.1. Revised Section 11.6.1 to read "Prior to concentrating each sample, add 100 uL of water."

19.3.2. Revised Section 11.6.2 to read "Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 100 uL of water remains.

11.6.2.1 This blow down must take a minimum of 3.5 hours.

11.6.2.2 Extracts can not remain in the water bath longer than 5 minutes once concentrated."

19.3.3. Revised Section 11.6.3 to read "Add 400 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water."

19.3.4. Revised Section 11.11.1 to read "Prior to concentrating each sample, add 200 uL of water."

19.3.5. Revised Section 11.11.2 to read "Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 200 uL of water remains."

11.11.2.1 This blow down must take a minimum of 3.5 hours.

11.11.2.2 Extracts can not remain in the water bath longer than 5 minutes once concentrated."

19.3.6. Revised Section 11.11.3 to read "Add 800 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water."

19.4. WS-LC-0025, Revision 2.4, Effective 04/25/2017

19.4.1. Removed all references to Method ISO 25101from this SOP due to the creation of WS-DW-0005 which covers Method ISO 25101.

19.4.2. Section 7.6, changed the concentration from 500 ng/mL to 20 ng/mL to increase spike amounts by diluting the solution in order to reduce the percent error when spiking.

19.4.3. Section 7.7, changed the concentration from 1000 ng/mL to 50 ng/mL to increase spike amounts by diluting the solution in order to reduce the percent error when spiking.

19.4.4. Section 11.2.5, changed 0.020mL (20uL) to 0.5 mL and changed 200 uL to 2.5 mL to aid in spiking efficiency.

19.4.5. Section 11.2.6, changed 0.025 mL (25 uL) to 0.5 mL and changed 125 uL to 2.5 mL to aid in spiking efficiency.

19.4.6. Ection 11.2.7, added "QC samples, LCS, MS, and MSD will require concentration via nitrogen to adjust the FC to 5.0 mL" to adjust for the increased spike volumes.

19.4.7. Section 11.7.4, changed 0.040 (40 uL) to 1.0 mL to aid in spiking efficiency.

19.4.8. Section 11.7.5, changed 0.05 mL (50 uL) to 1.0 mL to aid in spiking efficiency.

19.4.9. Editorial changes.

19.5. WS-LC-0025, Revision 2.3, Effective 04/10/2017

19.5.1. Updated the title to include Method ISO 25101:2009.

19.5.2. Removed Section 1.3," Due to poor chromatographic peak shape which degraded with repeated injections for Perfluoro-1-octanesulfonamidoamide (FOSSA), this analyte is no longer included in the method.", as this no longer applies. Renumbered subsections in Section 1.

19.5.3. Inserted Section 1.4, "This procedure also includes direction for preparing and analyzing samples to determine "Total Oxidizable Precursors", which may assist in improving understanding of potential PFAS environmental risk."

19.5.4. Added Section 2.6 to read, "Samples for the "Total Oxidizable Precursor" assay (TOP) are analyzed in two phases – an aliquot is prepared and analyzed as a normal sample, and a second aliquot is subjected to oxidation with potassium persulfate and sodium hydroxide prior to solid phase extraction and analysis. The total perfluorocarboxylic acid value is determined for each aliquot, and the difference calculated."

19.5.5. Changed all mentions of "direct aqueous injection (DAI)" to "in line solid phase extraction (SPE)."

19.5.6. Added Section 4.8 to read, "Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.15 pg/L or 0.01 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

19.5.7. Section 5.2, added "Potassium Persulfate (2-0-1-OX)" to the table.

19.5.8. Section 6, added the following:

- 125 mL HDPE containers with screw caps
- Water bath: Heated with concentric ring cover capable of temperature control (±5°C) up to 95°C. The bath must be used in a fume hood.
- Plastic tub for an ice bath, AKRO-N.S.T. part No. 35-180 or equivalent
- pH indicator paper, wide range

19.5.9. Section 7.1, added the following:

- Potassium persulfate, reagent grade
- Sodium hydroxide (NaOH), 10N, reagent grade
- Hydrochloric acid (HCl), concentrated, reagent grade

19.5.10. Added Section 7.2.1.1 to read, "Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.15 pg/L or 0.01 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

19.5.11. Section 7.4, added M2-4:2FTS to the "Initial Calibration Levels" table.

19.5.12. Added section 7.8 title, "Reverse Surrogate Solution, 1000 ng/mL" to read, "The reverse surrogate solution is prepared by diluting M2-4:2 FTS to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all samples for the TOP assay to monitor the efficiency of the oxidation process."

19.5.13. Added Section 11.12 titled, “Product Dispersion Samples” to read:

“11.12.1. Check the solubility of the material in both methanol and water

11.12.1.1 If the material is soluble in water, dilute 0.5 mL of sample into 250 mL of DI water and proceed to Section 11.3 (follow water extraction procedures). Fortify sample appropriately with IDA or PFC spike solution, see Section 11.2.

11.12.1.2 If the material is soluble in methanol, dilute 1 g (if solid) or 1 mL (if liquid) of material into 10 mL of methanol (MeOH).

11.12.1.2.1 If the material does not completely dissolve, contact your immediate supervisor.

11.12.2 Take 100 uL of the 10 mL solution and dilute it to 10 mL in MeOH.

11.12.3 Take a 1 mL aliquot of this solution (effective dilution of 1000x (1 mg for solid or 0.001 mL for liquid)) and fortify with 25 uL of labeled IDA or surrogate solution (Section 7.7).

11.12.4 DO NOT PASS EXTRACT THROUGH SPE CARTIRIDGE (omit steps 11.9 – 11.11).

11.12.5 Proceed to Section 11.6 of this SOP for extract concentration.”

19.5.14. Added Section 11.13 titled, “ TOP (Total Oxidizable Precursor) Assay” to read:

11.13.1 Prepare 3-250 mL HDPE containers with HPLC grade water to create the needed QC Samples (MB, LCS/LCSD).

11.13.2 Prepare enough 125 mL HDPE containers as needed for all “Pre” and “Post” samples, including QC. Label each appropriately.

11.13.3 Spike the “Pre” and “Post” MB 125 mL containers with 25 uL of the reverse surrogate solution of M2-4:2 FTS (Section 7.8).

11.13.4 Spike the “Pre” and “Post” LCS/LCSD 125 mL containers with 20 uL of the LCS Spike solution (Section 7.6), both regular and “add-on”, and 25 uL of the reverse surrogate solution (Section 7.8).

11.13.5 Remove the methanol solvent from all Post QC sample 125 mL containers (MB and LCS/LCSD) by using N2 evaporation.

11.13.6 Subsample 100 mL aliquots of water from each field sample and QC from the 250 mL containers into each of the corresponding 125 mL containers for both the “Pre” and “Post” samples.

11.13.7 Set aside all “Pre” sample containers.

11.13.8 Add 2g of potassium persulfate and 1.9 mL of 10N NaOH to each “Post” sample container.

11.13.9 Heat each “Post” sample container in a water bath (KD) at 85°C for 6 hours.

11.13.10 After digestion for 6 hours, place the “Post” sample containers in an ice bath for 30 minutes.

11.13.11 Adjust the pH of “Post” samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.13.12 Spike both “Pre” and “Post” samples and their associated QC samples with 25 uL of PFC IDA solution (Section 7.7), both regular and add-on.

11.13.13 Use the following SPE procedure for both “Pre” and “Post” samples:

11.13.13.1 Set up WAX 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.13.13.2 Establish a sample loading flow rate of 1 mL/minute for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.13.13.3 Wash/condition the SPE column with 5 mL of 0.3% NH_4OH/Methanol, then 5 mL water.

11.13.13.4 Load 100 mL of sample onto the SPE cartridge at a flow rate of 1 mL/minute.

11.13.13.5 Add 5 mL rinse water

11.13.13.6 After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.13.13.7 Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.13.13.8 Allow the column to dry well using vacuum with a flow rate of 1 mL/minute for 5 minutes. Columns must be dry before continuing.

11.13.13.9 Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 5 mL of 0.3% NH_4OH/methanol, and add to the SPE cartridge as eluent.

11.13.13.10 Repeat with another 5 mL of 0.3% NH_4OH/methanol.

11.13.13.11 Collect the 10 mL of eluent and concentrate per Section 11.6.

19.5.15. Section 11.15, added M2-4:2FTS to the "Mass Spectrometer Scan Settings (SCIEX 5500)" table.

19.5.16. Inserted Section 16.8 to read, "Method ISO 25101, "Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry", First Edition, 2009-03-01, International Organization for Standardization, Technical Committee ISO/TC 147, *Water Quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*."

19.5.17. Added Section 16.10 to read, "Erika F. Houtz and David L. Sedlak, "Oxidative Conversion as a Means of Detecting Precursors to Perfluoroalkyl Acids in Urban Runoff," Environmental Science and Technology 46, no. 17 (2012): 9342-49."

19.5.18. Section 17, inserted Section 17.1, and placed all modifications to Method 537 under Section 17.2 and subheadings.

19.5.19. Editorial changes.

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water samples via in line solid phase extraction (SPE) for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500.

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1

1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	1.0 mL	2.0 ng/L	2 to 200 ng/L

2. SUMMARY OF METHOD

2.1. A 1 mL aliquot of sample is diluted to a 40:60 methanol:water extract and analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using 20mM ammonium acetate/water and methanol.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of definitions.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for safety information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for supplies, other than those listed below specific to the in line SPE analysis.

6.1. 2 mL auto sampler vials, clear glass, Thermo Scientific Nation surestop vial, part no. C5000-1, or equivalent**.**

6.2. Vial caps, Thermo Scientific National AVCS blue cap, pre slit TEF/STL septa, part no. C5000-55B or equivalent.

6.3. Eppendorf 1000 uL epTIPS, part no. 022491954 or equivalent.

6.4. Eppendorf 200 uL epTIPS, part no. 022491938 or equivalent.

6.5. 50 mL graduated plastic centrifuge tubes, SCP Science DigiTUBES part no. 010-500-263 or equivalent

6.6. 1000 uL Pipette: Eppendorf Research Plus

6.7. 100 uL Pipette: Rainin EDP3-Plus

6.8. 250 mL HDPE bottles with PPE screw caps, ESS part no. 0250-1902-QC or equivalent.

6.9. Analytical columns

6.9.1. Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.

6.9.2. PFAS Isolator column, Phenomenex Luna C18 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E 0 or equivalent.

6.10. SCIEX 5500 Triple Quad MS. The system utilizes Chrom Peak Review, version 2.1 or equivalent.

6.11. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

7. REAGENTS AND STANDARDS

Refer to Section 7 of the main body of this SOP for reagents and standards, other than those listed below specific to the in line SPE analysis.

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Ammonium acetate, Fisher Optima LCMS grade (20 mM in water), part no. A114-50, or equivalent.

7.1.2. Methanol, Baker HPLC grade, part no. 9093-03.

7.1.3. Water, Nanopure or Millipore or Fisher Optima LCMS grade, part no. W6-4, must be free of interference and target analytes.

7.2. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of the stock solutions (Section 7.2 of the main body of this SOP) in 40:60 methanol:water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.3. Initial Calibration (ICAL) Levels (ng/L)

Compound	**CS-1**	**CS-2**	**CS-3**	**CS-4**	**CS-5**	**CS-6**	**CS-7**	**CS-8**
Perfluoroalkylcarboxylic acids (PFCAs)								
PFHpA	1.0	2.0	5.0	10	20	50	100	200
PFOA	1.0	2.0	5.0	10	20	50	100	200
PFNA	1.0	2.0	5.0	10	20	50	100	200
Perfluorinated sulfonic acids (PFSAs)								
PFBS	1.0	2.0	5.0	10	20	50	100	200
PFHxS	1.0	2.0	5.0	10	20	50	100	200
PFOS	1.0	2.0	5.0	10	20	50	100	200
Labeled Isotope Dilution Analytes (IDA)								
13C4-PFHpA	50	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50	50

Note- The above calibration levels are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.

7.4. LCS/Matrix PFC Spike Solution, 100 ng/mL.

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at 100 ng/mL in methanol.

7.5. PFC Isotope Dilution Analyte (IDA) Spike Solution, 1 ng/mL.

The PFC-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each at 1 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6 °C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6 °C. Water samples must be analyzed within 28 days of collection.

9. QUALITY CONTROL

Refer to Section 9 of the main body of this SOP for Quality Control information.

9.1. If potable water samples from the state of New York (NY) are analyzed via this method the control limits for LCS and IDA for PFOS and PFOA recoveries are 70-130%. If these limits are not met, refer to Section 9 of the main body of this SOP for corrective action.

9.2. If POST (treatment) samples have positive detections, review the associated PRE and MID (treatment) samples for similar detections. Re-preparation and re-analysis may be needed.

9.3. If PFBS is detected in the method blank greater than the RL, evaluate data for impact. PFBS is a known laboratory artifact. Re-preparation and re-analysis may be needed.

10. CALIBRATION

Refer to Section 10 of the main body of the SOP for calibration information.

11. PROCEDURE

Refer to Section 11 of the main body of this SOP for procedures, other than those listed below specific to the in line SPE analysis.

11.1. Water Sample Preparation

11.1.1. Visually inspect samples for the presence of settled and or suspended sediment/particulate. Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.

If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity of vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.1.2. Prepare an LCS and method blank by adding 250 mL of HPLC grade water into a 250 mL HDPE bottle.

11.1.3. If requested, find the client assigned sample for MS/MSD.

11.1.4. Spike directly into the sample bottles for the LCS and MS/MSD (if requested) with 0.050 mL (50 uL) of the LCS/Matrix PFC Spike solution (Section 7.4). This will result in a sample concentration of 20 ng/L. Shake well to disperse spike.

11.1.5. Measure 1 mL of each sample using an Eppendorf pipette and pour into a labeled 2.0 mL injection vial. This includes the LCS and method blank samples as well.

11.1.6. Be sure to “prepare” the pipette by collecting two 1 mL aliquots and disposing of them, and then collect the aliquot for testing.

11.1.7. Add 83 uL of surrogate solution (PFC IDA Spike Solution, Section 7.5) into each vial for each sample and QC sample. This will result in an extract concentration of 50 ng/L for the surrogate.

11.1.8. Add 577 uL of methanol to each sample for a final solvent composition of 40:60 methanol:water.

11.1.9. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.1.10. Vortex to mix the mixture well.

11.2. Instrument Analysis

11.2.1. Suggested operation conditions are listed below:

Routine Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
Column (Column temp = 35°C)	Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water	B = Methanol			
Gradient Program	**Time (min)**	**%A**	**%B**	**Curve**	**Flow Rate (mL/min)**
	0	90	10	6	0.60
	1	90	10	6	0.60
	1.5	35	65	6	0.60
	8	5	95	6	0.60
	8.1	1	99	6	0.60
	12	1	99	6	0.60
	12.5	90	10	6	0.60
	Maximum Pressure limit = 5,000 psi				
Injection Size	950 uL (fixed amount throughout the sequence)				
Run Time	17.1 minutes				

Routine Instrument Operating Conditions	
Mass Spectrometer Interface Settings (SCIEX 5500)	
MS Interface Mode	ESI Negative Ion
Ion Spray Voltage (kV)	4.5
Entrance Potential (V)	5
Declustering Potential (V)	25
Desolvation Temp	550 ºC
Curtain Gas (nitrogen) Flow	35 psi
Collision Gas (nitrogen) Flow	8 psi

Routine Instrument Operating Conditions						
Mass Spectrometer Scan Settings (SCIEX 5500)						
Compound	**Comments**	**Reaction (MRM)**	**Dwell (sec)**	**Ent. Pot. (V)**	**Col. Energy (V)**	**Declu. Pot. (V)**
PFBS	Perfluorobutanesulfonate	299 > 80	0.02	6	58	55
18O2-PFHxS	IDA	403 > 84	0.02	12	74	60
PFHpA	Perfluoroheptanoic acid	363 > 319	0.02	6	12	25
13C4-PFHpA	IDA	367 > 322	0.02	6	12	25
PFHxS	Perfluorohexanesulfonate	399 > 80	0.02	12	74	60
18O2-PFHxS	IDA	403 > 84	0.02	12	74	60
PFOA	Perfluorooctanoic acid	413 > 369	0.02	6	14	25
13C4PFOA	IDA	417 > 372	0.02	6	14	25
PFNA	Perfluorononanoic acid	463 > 419	0.02	6	14	25
13C5-PFNA	IDA	468 > 423	0.02	6	14	25
PFOS	Perfluorooctanesulfonate	499 > 80	0.02	9	108	65
13C4-PFOS	IDA	503 > 80	0.02	9	108	65

Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBS	6.68	18O2-PFHxS	7.76	Isotope Dilution
PFHpA	7.77	13C4-PFHpA	7.77	Isotope Dilution
PFHxS	7.76	18O2-PFHxS	7.76	Isotope Dilution
PFOA	8.44	13C4-PFOA	8.44	Isotope Dilution
PFNA	9.10	13C5-PFNA	9.10	Isotope Dilution
PFOS	9.06	13C4-PFOS	9.06	Isotope Dilution

11.2.2. Tune and calibrate the instrument as described in Section 10.

12. CALCULATIONS

Refer to Section 12 of the main body of this SOP for calculation information.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance information.

14. POLLUTION PREVENTION

Refer to Section 14 of the main body of this SOP for pollution prevention information.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for waste management information.

16. REFERENCES

Refer to Section 16 of the main body of this SOP for reference information.

17. METHOD MODIFICATIONS

17.1. Refer to Section 17 of the main body of this SOP for modifications from Method 537, except as detailed below:

17.1.1. Water samples are prepared at 1.0 mL, not 250 mL.

17.1.2. Water sample containers are not preserved with Trizma. Holding time has been changed to 28 days for analysis.

17.1.3. The eluents and HPLC configuration differs. As a result the final extract is in 40:60 methanol:water.

18. ATTACHMENTS

There are no attachments to this Appendix.

19. REVISION HISTORY

Revisions prior to 04/10/2017 have been removed and are available in previous versions of this SOP.

19.1. WS-LC-0025, Attachment 1, Revision 2.7, Effective 09/22/2017

19.1.1. Section 6.5, removed "The 5 items above are to be maintained in the drawer labeled "Segregated Supplies for in line SPE Analysis" in the LC/MS instrument room."

19.1.2. Added Sections 9.1 – 9.3.

19.1.3. Updated Section 11.1.

19.1.4. Editorial changes.

19.2. WS-LC-0025 Attachment 1, Revision 2.6, Effective 08/11/2017

19.2.1. No revisions to this attachment.

19.3. WS-LC-0025 Attachment 1, Revision 2.5, Effective 07/10/2017

19.3.1. No revisions to this attachment.

19.4. WS-LC-0025 Attachment 1, Revision 2.4, Effective 04/25/2017

19.4.1. No revisions to this attachment.

19.5. WS-LC-0025 Attachment 1, Revision 2.3, Effective 04/10/2017

19.5.1. Changed all mentions of “direct aqueous injection (DAI)” to “in line solid phase extraction (SPE).”

19.5.2. Inserted Section 17.1, and changed formatting of the modifications to Method 537 to Section 17.2 and subheadings.

Appendix C

LABORATORY ACCREDITATION BUREAU a division of A-S-B

Certificate of Accreditation

ISO/IEC 17025:2005 **Certificate Number L2468**

TestAmerica Sacramento

880 Riverside Parkway
West Sacramento CA 95605

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: January 20, 2018

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 20th of January 2017

*See the laboratory's Scope of Accreditation for details of accredited parameters
**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).
Form 403.14 – Rev 1 7/3/13

Certificate # L2468

Scope of Accreditation
For
TestAmerica Sacramento

880 Riverside Parkway
West Sacramento, CA 95605
Ms. Lisa Stafford
916-373-5600

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (LABPR 403 DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **TestAmerica Sacramento** to perform the following tests:

Accreditation granted through: **January 20, 2018**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silica
ICP-AES	EPA 6010B/6010C	Silicon
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
CVAAS	EPA 7470A	Mercury

Non-Potable Water		
Technology	**Method**	**Analyte**
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric	EPA 410.4	Chemical Oxygen Demand (COD)
LC/MS/MS	EPA 6850	Perchlorate
Colorimetric	EPA 7196A	Chromium (Hexavalent)
Probe	EPA 9040B/9040C	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Nitrate
Ion Chromatography	EPA 9056A/300.0	Nitrite
Ion Chromatography	EPA 9056A/300.0	Orthophosphate
Ion Chromatography	EPA 9056A/300.0	Sulfate
Titration	SM 2320B	Alkalinity
Gravimetric	SM 2540B	Solids, Total
Gravimetric	SM 2540C	Solids, Total Dissolved
Gravimetric	SM 2540D	Solids, Total Suspended
Colorimetric/Hydrolysis	EPA 353.2 Modified / WS-WC-0050	Nitrocellulose
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane

Certificate # L2468

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0011	1,4-Dioxane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Dithiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Benzothiazole
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfoxide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Chloropicrin
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Acetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	2-Chloroacetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Oxathiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Dimethyl Disulfide
GC-IT/MS	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/8290A/1613B	OCDD
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	OCDF
GC/HRMS	EPA 8290/8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDF

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5,triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)
HPLC/UV	EPA 8330A Modified /WS-LC-0010	Nitroguanidine
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-ethylperfluoro-1- octanesulfonamido)-ethanol (N-Et-FOSE)
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-Methylperfluoro-1- octanesulfonamido)-ethanol (N-Me-FOSE)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctane sulfonamide (EtFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctane sulfonamide (MeFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acid (MeFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctanoic acid (PFOA)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorododecanoic acid (PFDoDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroocatane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	**Method**	**Type**
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics
Solid Phase Extraction	EPA 3535A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B/5030C	Volatile Organic Compounds
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537	Perfluorooctanoic acid (PFOA)
LC/MS/MS	EPA 537	Perfluorooctane Sulfonic Acid(PFOS)
Preparation	**Method**	**Type**
Solid Phase Extraction	EPA 537	Perfluoro compounds in Drinking Water

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
CVAAS	EPA 7471A/7471B	Mercury
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric/Hydrolysis	EPA 353.2 Modified /WS-WC-0050	Nitrocellulose
LC/MS/MS	EPA 6850	Perchlorate
Probe	EPA 9045C/9045D	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Sulfate
Ion Chromatography	EPA 9056A/300.0	Nitrate
Ion Chromatography	EPA 9056A/300.0	Nitrite
Gravimetric	ASTM D2216	%Moisture
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline Range Organics (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Dithiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Benzothiazole
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfoxide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Chloropicrin
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Acetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	2-Chloroacetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Oxathiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Dimethyl Disulfide
GC/MS SIM	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	OCDD
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	OCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDF

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5,triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)
HPLC/UV	EPA 8330A Modified / WS-LC-0010	Nitroguanidine
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-ethylperfluoro-1- octanesulfonamido)-ethanol [N-Et-FOSE]
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-Methylperfluoro-1- octanesulfonamido)-ethanol [N-Me-FOSE]
LC/MS/MS	EPA 537 Modified / WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctane sulfonamide (EtFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctane sulfonamide (MeFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acide (MeFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctanoic acid (PFOA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorododecanoic acid (PFDoDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroocatane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168

Solid and Chemical Materials

Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	**Method**	**Type**
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Acid Digestion (Solid)	EPA 3050B	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics
Ultrasonic Extraction	EPA 3550B/3550C	Semivolatile and Non-Volatile Organics
Solvent Dilution	EPA 3580A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B	Volatile Organic Compounds
Purge and Trap	EPA 5035/5035A	Volatile Organic Compounds
Table / Jar Shake	WS-OP-0005	Chemical Warfare Degradates (in solid)
Microwave Extraction	EPA 3546	Semivolatile and Non-Volatile Organics
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure

Air and Emissions		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel

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Air and Emissions		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
Gravimetric	40CFR Part 50 App B	TSP (Total Suspended Particulate)
Gravimetric	40CFR Part 50 App J	PM10
GC/MS	EPA TO14A/TO15	1,1,1-Trichloroethane
GC/MS	EPA TO14A/TO15	1,1,2,2-Tetrachloroethane
GC/MS	EPA TO14A/TO15	1,1,2-Trichloroethane
GC/MS	EPA TO14A/TO15	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA TO14A/TO15	1,1-Dichloroethane
GC/MS	EPA TO14A/TO15	1,1-Dichloroethene
GC/MS	EPA TO14A/TO15	1,2,3-Trichlorobenzene
GC/MS	EPA TO14A/TO15	1,2,3-Trichloropropane
GC/MS	EPA TO14A/TO15	1,2,4-Trichlorobenzene
GC/MS	EPA TO14A/TO15	1,2,4-Trimethylbenzene
GC/MS	EPA TO14A/TO15	1,2-Dibromoethane
GC/MS	EPA TO14A/TO15	1,2-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,2-Dichloroethane
GC/MS	EPA TO14A/TO15	1,2-Dichloropropane
GC/MS	EPA TO14A/TO15	1,3,5-Trimethylbenzene
GC/MS	EPA TO14A/TO15	1,3-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,4-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,4-Dioxane
GC/MS	EPA TO14A/TO15	2-Butanone (MEK)
GC/MS	EPA TO14A/TO15	2-Chlorotoluene
GC/MS	EPA TO14A/TO15	2-Hexanone (MBK)
GC/MS	EPA TO14A/TO15	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA TO14A/TO15	4-Ethyltoluene
GC/MS	EPA TO14A/TO15	4-Isopropyltoluene
GC/MS	EPA TO14A/TO15	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA TO14A/TO15	Acetone
GC/MS	EPA TO14A/TO15	Acrolein
GC/MS	EPA TO14A/TO15	Allyl Chloride
GC/MS	EPA TO14A/TO15	Alpha Methyl Styrene
GC/MS	EPA TO14A/TO15	Benzene

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Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO14A/TO15	Benzyl Chloride
GC/MS	EPA TO14A/TO15	Bromodichloromethane
GC/MS	EPA TO14A/TO15	Bromoform
GC/MS	EPA TO14A/TO15	Bromomethane
GC/MS	EPA TO14A/TO15	Butadiene (1,3-Butadiene)
GC/MS	EPA TO14A/TO15	Butane
GC/MS	EPA TO14A/TO15	Carbon Disulfide
GC/MS	EPA TO14A/TO15	Carbon Tetrachloride
GC/MS	EPA TO14A/TO15	Chlorobenzene
GC/MS	EPA TO14A/TO15	Chlorodifluoromethane
GC/MS	EPA TO14A/TO15	Chloroethane
GC/MS	EPA TO14A/TO15	Chloroform
GC/MS	EPA TO14A/TO15	Chloromethane
GC/MS	EPA TO14A/TO15	cis-1,2-Dichloroethene
GC/MS	EPA TO14A/TO15	cis-1,3-Dichloropropene
GC/MS	EPA TO14A/TO15	Cyclohexane
GC/MS	EPA TO14A/TO15	Dibromochloromethane
GC/MS	EPA TO14A/TO15	Dibromomethane
GC/MS	EPA TO14A/TO15	Dichlorodifluoromethane
GC/MS	EPA TO14A/TO15	Ethyl Acetate
GC/MS	EPA TO14A/TO15	Ethylbenzene
GC/MS	EPA TO14A/TO15	Hexachlorobutadiene
GC/MS	EPA TO14A/TO15	Hexane
GC/MS	EPA TO14A/TO15	Isooctane (2,2,4- Trimethylpentane)
GC/MS	EPA TO14A/TO15	Isopropyl Alcohol
GC/MS	EPA TO14A/TO15	Isopropylbenzene
GC/MS	EPA TO14A/TO15	m & p Xylene
GC/MS	EPA TO14A/TO15	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA TO14A/TO15	Methylene Chloride
GC/MS	EPA TO14A/TO15	Naphthalene
GC/MS	EPA TO14A/TO15	n-Butanol
GC/MS	EPA TO14A/TO15	n-Butylbenzene
GC/MS	EPA TO14A/TO15	n-Heptane
GC/MS	EPA TO14A/TO15	n-Nonane
GC/MS	EPA TO14A/TO15	n-Octane
GC/MS	EPA TO14A/TO15	n-Propylbenzene
GC/MS	EPA TO14A/TO15	o-Xylene
GC/MS	EPA TO14A/TO15	Pentane
GC/MS	EPA TO14A/TO15	Propene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO14A/TO15	sec-Butylbenzene
GC/MS	EPA TO14A/TO15	Styrene
GC/MS	EPA TO14A/TO15	tert-Butylbenzene
GC/MS	EPA TO14A/TO15	Tetrachloroethene
GC/MS	EPA TO14A/TO15	Tetrahydrofuran
GC/MS	EPA TO14A/TO15	Toluene
GC/MS	EPA TO14A/TO15	trans-1,2-Dichloroethene
GC/MS	EPA TO14A/TO15	trans-1,3-Dichloropropene
GC/MS	EPA TO14A/TO15	Trichloroethene
GC/MS	EPA TO14A/TO15	Trichlorofluoromethane
GC/MS	EPA TO14A/TO15	Vinyl Acetate
GC/MS	EPA TO14A/TO15	Vinyl Bromide
GC/MS	EPA TO14A/TO15	Vinyl Chloride
GC/MS	EPA TO14A/TO15	Xylenes, Total
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Dioxide
GC-FID/TCD	ASTM1946D / EPA 3C	Nitrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Oxygen
GC-FID/TCD	ASTM1946D / EPA 3C	Helium
GC-FID/TCD	ASTM1946D / EPA 3C	Hydrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Methane
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Monoxide[A1]
GC/MS	EPA TO14A/TO15	Gasoline Range Organics (GRO)
GC/MS	EPA TO14A/TO15	TPH as Gasoline
GC/MS SIM	EPA TO15 SIM	1,1,1-Trichloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS SIM	EPA TO15 SIM	1,1-Dichloroethane
GC/MS SIM	EPA TO15 SIM	1,1-Dichloroethene
GC/MS SIM	EPA TO15 SIM	1,2,3-Trichloropropane
GC/MS SIM	EPA TO15 SIM	1,2,4-Trichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,2-Dibromoethane
GC/MS SIM	EPA TO15 SIM	1,2-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,2-Dichloroethane
GC/MS SIM	EPA TO15 SIM	1,2-Dichloropropane
GC/MS SIM	EPA TO15 SIM	1,3-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,4-Dioxane
GC/MS SIM	EPA TO15 SIM	Acrolein

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA TO15 SIM	Benzene
GC/MS SIM	EPA TO15 SIM	Benzyl Chloride
GC/MS SIM	EPA TO15 SIM	Bromodichloromethane
GC/MS SIM	EPA TO15 SIM	Butadiene (1,3-Butadiene)
GC/MS SIM	EPA TO15 SIM	Carbon Tetrachloride
GC/MS SIM	EPA TO15 SIM	Chlorobenzene
GC/MS SIM	EPA TO15 SIM	Chloroethane
GC/MS SIM	EPA TO15 SIM	Chloroform
GC/MS SIM	EPA TO15 SIM	Chloromethane
GC/MS SIM	EPA TO15 SIM	cis-1,2-Dichloroethene
GC/MS SIM	EPA TO15 SIM	cis-1,3-Dichloropropene
GC/MS SIM	EPA TO15 SIM	Dibromochloromethane
GC/MS SIM	EPA TO15 SIM	Dichlorodifluoromethane
GC/MS SIM	EPA TO15 SIM	Ethylbenzene
GC/MS SIM	EPA TO15 SIM	Hexachlorobutadiene
GC/MS SIM	EPA TO15 SIM	m & p Xylene
GC/MS SIM	EPA TO15 SIM	Methyl tert-butyl Ether (MTBE)
GC/MS SIM	EPA TO15 SIM	Methylene Chloride
GC/MS SIM	EPA TO15 SIM	Naphthalene
GC/MS SIM	EPA TO15 SIM	o-Xylene
GC/MS SIM	EPA TO15 SIM	Styrene
GC/MS SIM	EPA TO15 SIM	Tetrachloroethene
GC/MS SIM	EPA TO15 SIM	Toluene
GC/MS SIM	EPA TO15 SIM	trans-1,2-Dichloroethene
GC/MS SIM	EPA TO15 SIM	trans-1,3-Dichloropropene
GC/MS SIM	EPA TO15 SIM	Trichloroethene
GC/MS SIM	EPA TO15 SIM	Trichlorofluoromethane
GC/MS SIM	EPA TO15 SIM	Vinyl Chloride
GC/MS SIM	EPA TO15 SIM	Xylenes, Total
GC/MS	EPA TO-13A	1,2,4-Trichlorobenzene
GC/MS	EPA TO-13A	1,2-Dichlorobenzene
GC/MS	EPA TO-13A	1,3-Dichlorobenzene
GC/MS	EPA TO-13A	1,3-Dinitrobenzene
GC/MS	EPA TO-13A	1,4-Dichlorobenzene
GC/MS	EPA TO-13A	1-Methylnaphthalene
GC/MS	EPA TO-13A	2,3,4,6-Tetrachlorophenol
GC/MS	EPA TO-13A	2,4,5-Trichlorophenol
GC/MS	EPA TO-13A	2,4,6-Trichlorophenol
GC/MS	EPA TO-13A	2,4-Dichlorophenol

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Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-13A	2,4-Dimethylphenol
GC/MS	EPA TO-13A	2,4-Dinitrophenol
GC/MS	EPA TO-13A	2,4-Dinitrotoluene
GC/MS	EPA TO-13A	2,6-Dichlorophenol
GC/MS	EPA TO-13A	2,6-Dinitrotoluene
GC/MS	EPA TO-13A	2-Chloronaphthalene
GC/MS	EPA TO-13A	2-Chlorophenol
GC/MS	EPA TO-13A	2-Methylnaphthalene
GC/MS	EPA TO-13A	2-Methylphenol
GC/MS	EPA TO-13A	2-Nitroaniline
GC/MS	EPA TO-13A	2-Nitrophenol
GC/MS	EPA TO-13A	3&4-Methylphenol
GC/MS	EPA TO-13A	3,3'-Dichlorobenzidine
GC/MS	EPA TO-13A	3-Nitroaniline
GC/MS	EPA TO-13A	4,6-Dinitro-2-methylphenol
GC/MS	EPA TO-13A	4-Bromophenyl phenyl ether
GC/MS	EPA TO-13A	4-Chloro-3-methylphenol
GC/MS	EPA TO-13A	4-Chloroaniline
GC/MS	EPA TO-13A	4-Chlorophenyl phenyl ether
GC/MS	EPA TO-13A	4-Nitroaniline
GC/MS	EPA TO-13A	4-Nitrophenol
GC/MS	EPA TO-13A	Acenaphthene
GC/MS	EPA TO-13A	Acenaphthylene
GC/MS	EPA TO-13A	Aniline
GC/MS	EPA TO-13A	Anthracene
GC/MS	EPA TO-13A	Benzo(a)anthracene
GC/MS	EPA TO-13A	Benzo(a)pyrene
GC/MS	EPA TO-13A	Benzo(b)fluoranthene
GC/MS	EPA TO-13A	Benzo(g,h,i)perylene
GC/MS	EPA TO-13A	Benzo(k)fluoranthene
GC/MS	EPA TO-13A	Benzoic Acid
GC/MS	EPA TO-13A	Benzyl Alcohol
GC/MS	EPA TO-13A	Benzyl butyl Phthalate
GC/MS	EPA TO-13A	Biphenyl
GC/MS	EPA TO-13A	Bis(2-chloroethoxy) Methane
GC/MS	EPA TO-13A	Bis(2-chloroethyl) Ether
GC/MS	EPA TO-13A	Bis(2-chloroisopropyl) Ether
GC/MS	EPA TO-13A	Carbazole
GC/MS	EPA TO-13A	Chrysene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-13A	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA TO-13A	Dibenz(a,h)anthracene
GC/MS	EPA TO-13A	Dibenzofuran
GC/MS	EPA TO-13A	Diethyl Phthalate
GC/MS	EPA TO-13A	Dimethyl Phthalate
GC/MS	EPA TO-13A	Di-n-butyl Phthalate
GC/MS	EPA TO-13A	Di-n-octyl Phthalate
GC/MS	EPA TO-13A	Fluoranthene
GC/MS	EPA TO-13A	Fluorene
GC/MS	EPA TO-13A	Hexachlorobenzene
GC/MS	EPA TO-13A	Hexachlorobutadiene
GC/MS	EPA TO-13A	Hexachlorocyclopentadiene
GC/MS	EPA TO-13A	Hexachloroethane
GC/MS	EPA TO-13A	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA TO-13A	Isophorone
GC/MS	EPA TO-13A	Naphthalene
GC/MS	EPA TO-13A	Nitrobenzene
GC/MS	EPA TO-13A	n-Nitrosodimethylamine
GC/MS	EPA TO-13A	n-Nitrosodi-n-propylamine
GC/MS	EPA TO-13A	n-Nitrosodiphenylamine
GC/MS	EPA TO-13A	Pentachlorophenol
GC/MS	EPA TO-13A	Phenanthrene
GC/MS	EPA TO-13A	Phenol
GC/MS	EPA TO-13A	Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	1-Methylnaphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	2-Methylnaphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(b)fluoranthene

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Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(g,h,i)perylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(k)fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Chrysene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluorene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Naphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Phenanthrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Pyrene
GC-ECD	EPA TO-4A/TO-10A	PCB-1016
GC-ECD	EPA TO-4A/TO-10A	PCB-1221
GC-ECD	EPA TO-4A/TO-10A	PCB-1232
GC-ECD	EPA TO-4A/TO-10A	PCB-1242
GC-ECD	EPA TO-4A/TO-10A	PCB-1248
GC-ECD	EPA TO-4A/TO-10A	PCB-1254
GC-ECD	EPA TO-4A/TO-10A	PCB-1260
GC-ECD	EPA TO-4A/TO-10A	PCB-1262
GC-ECD	EPA TO-4A/TO-10A	PCB-1268
Preparation	**Method**	**Type**
Acid Digestion (Filters, Solid)	EPA 3050B	Inorganics
Soxhlet extraction of PUF	TO-4A/TO-10A	PCBs in Air
Soxhlet extraction of PUF/XAD	TO-13	Semivolatiles in Air
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs

Certificate # L2468

Notes:

1) This laboratory offers commercial testing service.

Approved by: ______________________ Date: January 20, 2017

R. Douglas Leonard
Chief Technical Officer

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